

## PATENT COOPERATION TREATY

PCT

## NOTIFICATION OF ELECTION

(PCT Rule 61.2)

From the INTERNATIONAL BUREAU

To:

Assistant Commissioner for Patents  
 United States Patent and Trademark  
 Office  
 Box PCT  
 Washington, D.C.20231  
 ETATS-UNIS D'AMERIQUE

in its capacity as elected Office

Date of mailing (day/month/year)

15 August 2000 (15.08.00)

International application No.

PCT/SE99/02170

Applicant's or agent's file reference

P15634PC/SC

International filing date (day/month/year)

24 November 1999 (24.11.99)

Priority date (day/month/year)

26 November 1998 (26.11.98)

Applicant

SANDQUIST, Kent, K. et al

1. The designated Office is hereby notified of its election made:



in the demand filed with the International Preliminary Examining Authority on:

13 June 2000 (13.06.00)



in a notice effecting later election filed with the International Bureau on:

2. The election



was



was not

made before the expiration of 19 months from the priority date or, where Rule 32 applies, within the time limit under Rule 32.2(b).

The International Bureau of WIPO  
 34, chemin des Colombettes  
 1211 Geneva 20, Switzerland

Facsimile No.: (41-22) 740.14.35

Authorized officer

F. Baechler

Telephone No.: (41-22) 338.83.38

# PATENT COOPERATION TREATY

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## INTERNATIONAL SEARCH REPORT

International application No.

PCT/SE 99/02170

## A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D21C 11/08, D21C 11/06

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC7: D21C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EDOC, WPI, FULLTEXT

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 1354499 A (CHEMECH ENGINEERING LTD.), 30 May 1974 (30.05.74)	1-11
A	WO 8600389 A1 (HEED, BJÖRN), 16 January 1986 (16.01.86)	1-11
P,A	WO 9855685 A1 (THERMATRIX INC.), 10 December 1998 (10.12.98)	1-11

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

\* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"I" earlier document but published on or after the international filing date

"I" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&amp;" document member of the same patent family

Date of the actual completion of the international search

10 March 2000

Name and mailing address of the ISA/

Swedish Patent Office

Box 5055, S-102 42 STOCKHOLM

Facsimile No. +46 8 666 02 86

Date of mailing of the international search report

14 -03- 2000

Authorized officer

Helena Hemphälä/ELY

Telephone No. +46 8 782 25 00

# INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/SE 99/02170

Patent document cited in search report			Publication date	Patent family member(s)		Publication date
GB	1354499	A	30/05/74	CA	926561 A	22/05/73
WO	8600389	A1	16/01/86	AT	41052 T	15/03/89
				CA	1249213 A	24/01/89
				DE	3590307 T	04/06/87
				EP	0218590 A,B	22/04/87
				SE	0218590 T3	
				JP	7033905 B	12/04/95
				JP	61502484 T	30/10/86
				SE	441623 B,C	21/10/85
				SE	8403330 D	00/00/00
				US	4741690 A	03/05/88
WO	9855685	A1	10/12/98	AU	7702698 A	21/12/98
				EP	0958726 A	24/11/99

## PATENT COOPERATION TREATY

## PCT

REC'D 11 JAN 2001

WIPO

PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference P15634PC/SC	<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. PCT/SE99/02170	International filing date (day/month/year) 24/11/1999	Priority date (day/month/year) 26/11/1998
International Patent Classification (IPC) or national classification and IPC D21C11/08		
Applicant EXCELENTEC HOLDING AB et al.		

1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.



2. This REPORT consists of a total of 4 sheets, including this cover sheet.

- ☐ This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).

These annexes consist of a total of sheets.

3. This report contains indications relating to the following items:

- I ☒ Basis of the report
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☒ Certain observations on the international application

Date of submission of the demand 13/06/2000	Date of completion of this report 08.01.2001
Name and mailing address of the international preliminary examining authority:  European Patent Office D-80298 Munich Tel. +49 89 2399 - 0 Tx: 523656 epmu d Fax: +49 89 2399 - 4465	Authorized officer Tragoustis, M Telephone No. +49 89 2399 8623 

# INTERNATIONAL PRELIMINARY EXAMINATION REPORT

International application No. PCT/SE99/02170

## I. Basis of the report

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).)*:

### Description, pages:

1-8 as originally filed

### Claims, No.:

1-11 as originally filed

### Drawings, sheets:

1/1 as originally filed

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/SE99/02170

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes: Claims 1-11
	No: Claims
Inventive step (IS)	Yes: Claims 1-11
	No: Claims
Industrial applicability (IA)	Yes: Claims 1-11
	No: Claims

- 2. Citations and explanations**  
**see separate sheet**

**VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:  
**see separate sheet**

1. The application relates to a method of removing sulphides and other volatile contaminants from liquor vapour condensate originating from a pulp manufacturing process.

According to claim 1 the sulphides are stripped off in a stripper from the condensate by a gas, which gas is then fed to a regenerative oxidation system, where the stripped gases are combusted and SO<sub>2</sub> is formed. The gas containing the SO<sub>2</sub> is then lead to a SO<sub>2</sub> scrubber and the gas from the scrubber is recirculated to the stripper.

By this closed loop of the gas, the cleaning of the condensates is carried out with high efficiency and good heat economy.

The documents of the Search Report are silent about the above three stages (stripping, oxidation, scrubbing) integrated in a closed loop cycle of the stripping gas.

WO 86/00389 discloses a process for combustion of pollutants carried in a gas. Sulfide contaminated condensates as well as stripping are not mentioned.

GB-A-1354499 discloses a process , whereby organic sulphur compounds present in condensates are stripped by an oxygen stream and are then catalytically oxidised. A closed loop for the stripping gas is not mentioned.

WO 98/55685 deals with the removal of sulphur from non-condensable gas stream released during the Kraft process.

Hence claim 1 and the dependent claims 2-11 meet the requirements of Art. 33 PCT.

2. Apparently (see also page 3,4 of the description) the SO<sub>2</sub> scrubber is part of the closed gas loop.

Hence the feature of claim 2 being an essential feature of the claimed invention should be included in claim 1 (Art. 6 PCT).

The word "either" in claim 1 causes unclarity and should be deleted.



# PCT

## REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.	PCT/SE99/02170
International Filing Date	24-11-1999
Name of receiving Office and "PCT International Application"	The Swedish Patent Office PCT International Application
Applicant's or agent's file reference (if desired) (12 characters maximum)	P15634PC/SC

<b>Box No. I TITLE OF INVENTION</b> A method for cleaning sulphide-containing condensates	
<b>Box No. II APPLICANT</b>	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.) EXELENTEC AB Bror Nilssons gata 16 S-417 55 GÖTEBORG Sweden	<input type="checkbox"/> This person is also inventor.  Telephone No.  Facsimile No.  Teleprinter No.
State (i.e. country) of nationality: Sweden	State (that is, country) of residence: Sweden
This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input checked="" type="checkbox"/> all designated States except the United States of America <input type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box	
<b>Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)</b>	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (i.e. country) of residence if no State of residence is indicated below.) SANDQUIST, KENT K Askims Fornborgsv. 2 S-436 42 ASKIM Sweden	This person is: <input type="checkbox"/> applicant only <input checked="" type="checkbox"/> applicant and inventor <input type="checkbox"/> inventor only (If this check-box is marked, do not fill in below.)
State (that is, country) of nationality: Sweden	State (that is, country) of residence: Sweden
This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input checked="" type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box	
<input checked="" type="checkbox"/> Further applicants and/or (further) inventors are indicated on a continuation sheet.	
<b>Box No. IV AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE</b>	
The person identified below is hereby/has been appointed to act on behalf of the applicant(s) before the competent International Authorities as: <input checked="" type="checkbox"/> agent <input type="checkbox"/> common representative	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.) GÖTEBORGS PATENTBYRÅ DAHLS AB Sjöporten 4 S-417 64 GÖTEBORG Sweden	Telephone No. +46-31-507700  Facsimile No. +46-31-7790640  Teleprinter No.
<input type="checkbox"/> Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.	

see #09

Sheet No. 2 -

Continuation of Box No. III FURTHER APPLICANTS AND/OR (FURTHER) INVENTORS	
If none of the following sub-boxes is used, this sheet is not to be included in the request.	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.) <b>WENNBERG, Olle</b> <b>Vaktmästaregången 1</b> <b>S-413 18 GÖTEBORG</b> <b>Sweden</b>	This person is: <input type="checkbox"/> applicant only <input checked="" type="checkbox"/> applicant and inventor <input type="checkbox"/> inventor only (if this check-box is marked, do not fill in below.)
State (that is, country) of nationality: <b>Sweden</b>	State (that is, country) of residence: <b>Sweden</b>
This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input checked="" type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)	This person is: <input type="checkbox"/> applicant only <input type="checkbox"/> applicant and inventor <input type="checkbox"/> inventor only (if this check-box is marked, do not fill in below.)
State (that is, country) of nationality:	State (that is, country) of residence:
This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)	This person is: <input type="checkbox"/> applicant only <input type="checkbox"/> applicant and inventor <input type="checkbox"/> inventor only (if this check-box is marked, do not fill in below.)
State (that is, country) of nationality:	State (that is, country) of residence:
This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box	
Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)	This person is: <input type="checkbox"/> applicant only <input type="checkbox"/> applicant and inventor <input type="checkbox"/> inventor only (if this check-box is marked, do not fill in below.)
State (that is, country) of nationality:	State (that is, country) of residence:
This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box	
<input type="checkbox"/> Further applicants and/or (further) inventors are indicated on another continuation sheet.	

## Box No. V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes: at least one must be marked):

## Regional Patent

- ☒ **AP ARIPO Patent:** GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SZ Swaziland, UG Uganda, ZW Zimbabwe and any other State which is a Contracting State of the Harare Protocol and of the PCT
- ☒ **EA Eurasian Patent:** AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- ☒ **EP European Patent:** AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- ☒ **OA OAPI Patent:** BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of PCT (if other kind of protection or treatment desired, specify on dotted line)-----

## National Patent (if other kind of protection or treatment desired specify on dotted line)

- |   |  |
|---|--|
| <input checked="" type="checkbox"/> <b>AE</b> United Arab Emirates                  | <input checked="" type="checkbox"/> <b>LR</b> Liberia  |
| <input checked="" type="checkbox"/> <b>AL</b> Albania                               | <input checked="" type="checkbox"/> <b>LS</b> Lesotho  |
| <input checked="" type="checkbox"/> <b>AM</b> Armenia                               | <input checked="" type="checkbox"/> <b>LT</b> Lithuania  |
| <input checked="" type="checkbox"/> <b>AT</b> Austria                               | <input checked="" type="checkbox"/> <b>LU</b> Luxembourg   |
| <input checked="" type="checkbox"/> <b>AU</b> Australia                             | <input checked="" type="checkbox"/> <b>LV</b> Latvia   |
| <input checked="" type="checkbox"/> <b>AZ</b> Azerbaijan                            | <input checked="" type="checkbox"/> <b>MD</b> Republic of Moldova  |
| <input checked="" type="checkbox"/> <b>BA</b> Bosnia and Herzegovina                | <input checked="" type="checkbox"/> <b>MG</b> Madagascar   |
| <input checked="" type="checkbox"/> <b>BB</b> Barbados                              | <input checked="" type="checkbox"/> <b>MK</b> The former Yugoslav Republic of Macedonia  |
| <input checked="" type="checkbox"/> <b>BG</b> Bulgaria                              |  |
| <input checked="" type="checkbox"/> <b>BR</b> Brazil                                | <input checked="" type="checkbox"/> <b>MN</b> Mongolia   |
| <input checked="" type="checkbox"/> <b>BY</b> Belarus                               | <input checked="" type="checkbox"/> <b>MW</b> Malawi   |
| <input checked="" type="checkbox"/> <b>CA</b> Canada                                | <input checked="" type="checkbox"/> <b>MX</b> Mexico   |
| <input checked="" type="checkbox"/> <b>CH and LI</b> Switzerland and Liechtenstein  | <input checked="" type="checkbox"/> <b>NO</b> Norway   |
| <input checked="" type="checkbox"/> <b>CN</b> China                                 | <input checked="" type="checkbox"/> <b>NZ</b> New Zealand  |
| <input checked="" type="checkbox"/> <b>CR</b> Costa Rica                            | <input checked="" type="checkbox"/> <b>PL</b> Poland   |
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| <input checked="" type="checkbox"/> <b>DE</b> Germany                               | <input checked="" type="checkbox"/> <b>RU</b> Russian Federation   |
| <input checked="" type="checkbox"/> <b>DK</b> Denmark                               | <input checked="" type="checkbox"/> <b>SD</b> Sudan  |
| <input checked="" type="checkbox"/> <b>DM</b> Dominica                              | <input checked="" type="checkbox"/> <b>SE</b> Sweden   |
| <input checked="" type="checkbox"/> <b>EE</b> Estonia                               | <input checked="" type="checkbox"/> <b>SG</b> Singapore  |
| <input checked="" type="checkbox"/> <b>ES</b> Spain                                 | <input checked="" type="checkbox"/> <b>SI</b> Slovenia   |
| <input checked="" type="checkbox"/> <b>FI</b> Finland                               | <input checked="" type="checkbox"/> <b>SL</b> Sierra Leone   |
| <input checked="" type="checkbox"/> <b>GB</b> United Kingdom                        | <input checked="" type="checkbox"/> <b>SK</b> Slovakia   |
| <input checked="" type="checkbox"/> <b>GD</b> Grenada                               | <input checked="" type="checkbox"/> <b>TJ</b> Tajikistan   |
| <input checked="" type="checkbox"/> <b>GE</b> Georgia                               | <input checked="" type="checkbox"/> <b>TM</b> Turkmenistan   |
| <input checked="" type="checkbox"/> <b>GH</b> Ghana                                 | <input checked="" type="checkbox"/> <b>TR</b> Turkey   |
| <input checked="" type="checkbox"/> <b>GM</b> Gambia                                | <input checked="" type="checkbox"/> <b>TT</b> Trinidad and Tobago  |
| <input checked="" type="checkbox"/> <b>HR</b> Croatia                               | <input checked="" type="checkbox"/> <b>TZ</b> United Republic of Tanzania  |
| <input checked="" type="checkbox"/> <b>HU</b> Hungary                               | <input checked="" type="checkbox"/> <b>UA</b> Ukraine  |
| <input checked="" type="checkbox"/> <b>ID</b> Indonesia                             | <input checked="" type="checkbox"/> <b>UG</b> Uganda   |
| <input checked="" type="checkbox"/> <b>IL</b> Israel                                | <input checked="" type="checkbox"/> <b>US</b> United States of America   |
| <input checked="" type="checkbox"/> <b>IN</b> India                                 |  |
| <input checked="" type="checkbox"/> <b>IS</b> Iceland                               | <input checked="" type="checkbox"/> <b>UZ</b> Uzbekistan   |
| <input checked="" type="checkbox"/> <b>JP</b> Japan                                 | <input checked="" type="checkbox"/> <b>VN</b> Viet Nam   |
| <input checked="" type="checkbox"/> <b>KE</b> Kenya                                 | <input checked="" type="checkbox"/> <b>YU</b> Yugoslavia   |
| <input checked="" type="checkbox"/> <b>KG</b> Kyrgyzstan                            | <input checked="" type="checkbox"/> <b>ZA</b> South Africa   |
| <input checked="" type="checkbox"/> <b>KP</b> Democratic People's Republic of Korea | <input checked="" type="checkbox"/> <b>ZW</b> Zimbabwe   |
| <input checked="" type="checkbox"/> <b>KR</b> Republic of Korea                     | Check-boxes reserved for designating States (for the purposes of a national patent) which have become party to the PCT after issuance of this sheet: |
| <input checked="" type="checkbox"/> <b>KZ</b> Kazakhstan                            | <input checked="" type="checkbox"/> <b>MA</b> Morocco  |
| <input checked="" type="checkbox"/> <b>LC</b> Saint Lucia                           | <input type="checkbox"/>   |
| <input checked="" type="checkbox"/> <b>LK</b> Sri Lanka                             |  |

**Precautionary Designation Statement:** In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. (Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit)

Box No. VI PRIORITY CLAIM				
<input type="checkbox"/> Further priority claims are indicated in the Supplemental Box.				
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country	regional application:* regional Office	international application: receiving Office
item (1) 26.11.98	9804061-1	Sweden		
item (2)				
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s): SE 9804061-1

\* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.

**Box No. VII INTERNATIONAL SEARCHING AUTHORITY**

<b>Choice of International Searching Authority (ISA)</b> (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):  <u>ISA/SE</u>	<b>Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority):</b>  Date (day/month/year)                      Number                      Country (or regional Office)
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**Box No. VIII CHECK LIST; LANGUAGE OF FILING.**

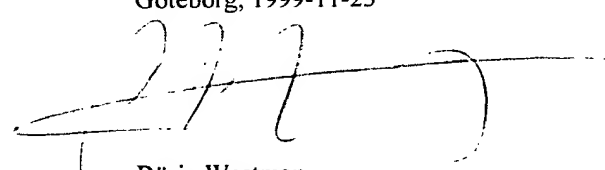
This international application contains the following number of sheets:  request : 4  description (excluding sequence listing part) : 8  claims : 3  abstract : 1  drawings : 1  sequence listing part of description :  Total number of sheets : 17	This international application is accompanied by the item(s) marked below: 1. <input checked="" type="checkbox"/> fee calculation sheet 2. <input checked="" type="checkbox"/> separate signed power of attorney 3. <input type="checkbox"/> copy of general power of attorney; reference number, if any: 4. <input type="checkbox"/> statement explaining lack of signature 5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s): 6. <input type="checkbox"/> translation of international application into (language): 7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material 8. <input type="checkbox"/> nucleotide and/or amino acid sequence listing in computer readable form 9. <input type="checkbox"/> other (specify):
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<b>Figure of the drawings</b> which should accompany the abstract: 1	<b>Language of filing of the international application:</b> Swedish
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**Box No. IX SIGNATURE OF APPLICANT OR AGENT**

Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).

Göteborg, 1999-11-23

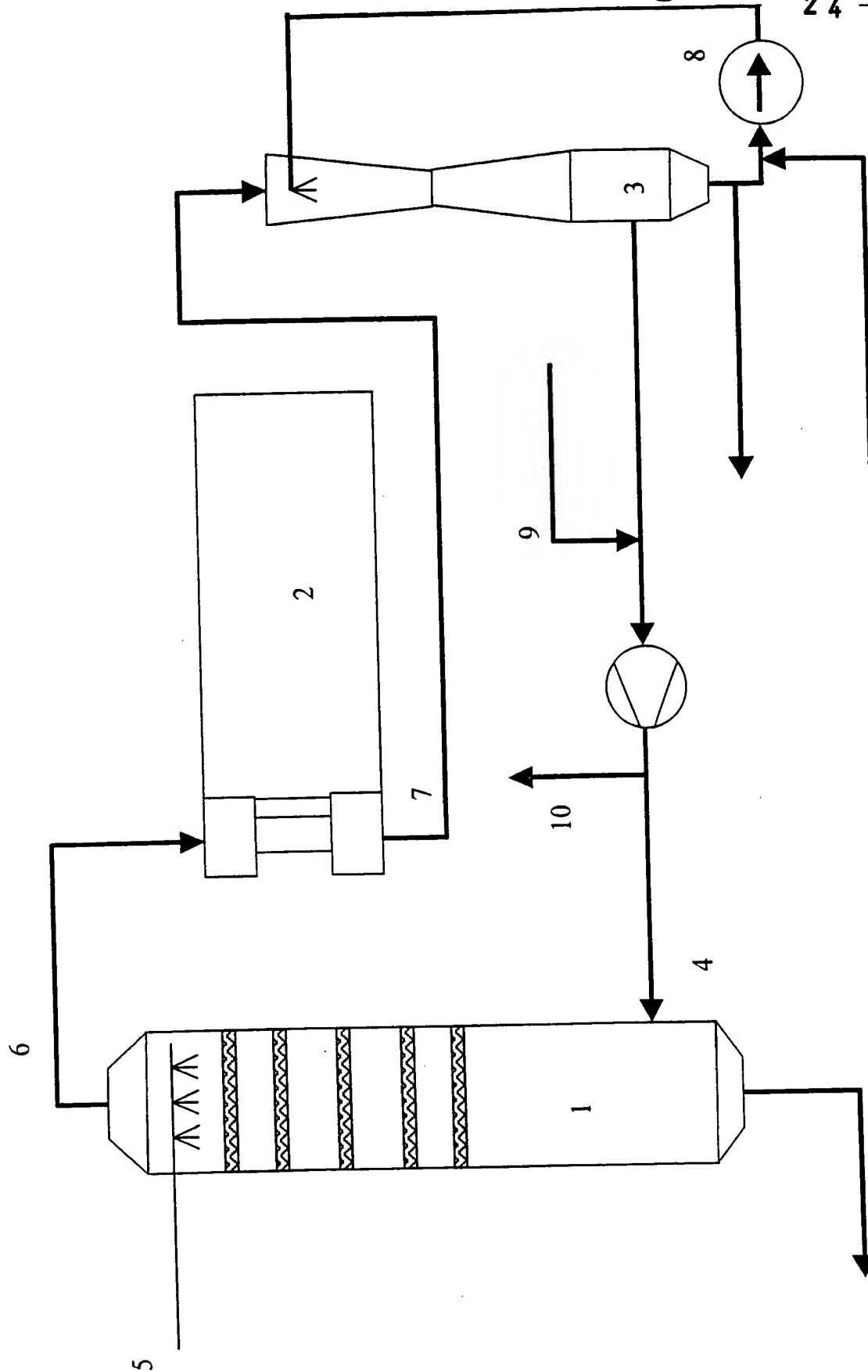


Börje Westman  
GÖTEBORGS PATENTBYRÅ DAHLS AB

For receiving Office use only		2. Drawings:  <input checked="" type="checkbox"/> received:  <input type="checkbox"/> not received:
1. Date of actual receipt of the purported international application:	24 -11- 1999	
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:		
4. Date of timely receipt of the required corrections under PCT Article 11(2):		
5. International Searching Authority (if two or more are competent): <u>ISA/SE</u>	6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid.	
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24 JANUARY 2000

FIG 1



# SÄTT ATT RENA SULFIDHALTIGA KONDENSAT

Vid framställning av cellulosa enligt sulfatprocessen avskiljes en avlut, som indunstas för att sedan förbrännas. Vid denna avdunstning avdrives lutånga, som förutom vattenånga innehåller diverse lättflyktiga föroreningar. Sådana föroreningar är vätesulfid, metylmerkaptan, dimetylsulfid, metanol, terpenier, m.m. I indunstningen som sker genom s.k. flereffektsindunstning med ett flertal steg, effekter (normalt 4-7 stycken), kondenseras lutången även den i ett flertal steg, varvid även stora delar av de lättflyktiga föroreningarna kondenserar. Kondensering sker i åtminstone lika många steg som det finns effekter. Detta medför att det erhålles starkt varierande kondensatkvalitéer från de olika stegen i indunstningen. Normalt urskiljes 2-3 olika kondensatkvalitéer, som var och en utgör en blandning av kondensat från olika steg. Det mest förorenade kondensatet (smutskondensat) behandlas normalt i en ångstripper, där de flyktiga komponenterna destilleras av. Detta smutskondensat utgör normalt endast en mindre del av hela kondensatmängden och därför belastas inte ångekonomin i någon högre utsträckning av att ånga används för strippningen. Investeringskostnaden för strippningen blir då också minimerad.

De övriga kondensatkvalitéernas renhet beror i hög utsträckning på andelen smutskondensat. Ökas andelen smutskondensat blir de andra kondensaten renare. En alltför stor smutskondensatmängd ger dock högre drifts- och kapitalkostnader för strippningen.

De andra, mindre förorenade kondensaten kan i viss utsträckning användas som processvatten beroende på deras renhet. Om kondensatet är alltför förorenat kan det däremot inte återanvändas utan måste i stället släppas ut till recipienten med eventuellt föregående rening.

Det som framför allt begränsar möjligheten att använda kondensat inom processen är innehållet av sulfider, eftersom dessa kan ge obehaglig lukt och smak hos pappersmassan. Detta utgör även ett betydande problem för arbetsmiljön. Terpen-  
5 ger också lukt. Terpenerna förekommer dock normalt i mycket låga halter i de mindre förorenade kondensaten.

Den teknik som står till buds för rening av dessa kondensat är huvudsakligen ångstrippning. Eftersom kondensatmängderna  
10 är mycket stora, krävs en stor stripperutrustning och stora mängder ånga för strippningen. Ångmängderna blir så stora att det definitivt inte kan vara ekonomiskt försvarbart att använda färskånga. Däremot kan man låta använda ångan, som drivs av vid indunstningen av avluten, i flereffekts-  
15 indunstning för strippning. Ångan som lämnar strippern kan sedan återvinnas som värme i nästföljande effekt. Reningsgraden för en sådan stripper är dock begränsad p.g.a. att lutången från den föregående effekten är förorenad vilket begränsar renheten i det utgående kondensatet. Framför allt  
20 begränsas renheten med avseende på sulfider, eftersom lutången kan ha ett betydande innehåll av sulfider. Detta sulfidinnehåll beror på att man normalt tar ånga från första effekten, där temperaturen är relativt hög, vilket ger en förhöjd sulfidhalt.

25 En ytterligare nackdel är att ångan vid passagen genom strippern tappar i tryck och att lättflyktiga komponenter anrikas. Dessa två saker medför att ångans kondenserings-temperatur minskar, vilket medför att den tillgängliga  
30 temperaturdifferensen i indunstningen minskar. Detta har en negativ inverkan både energi- och kapitalkostnaderna för indunstningsanläggningen. Dessutom är indunstningen och stripper helt integrerade, varför dessa två delar inte kan köras oberoende av varandra.

35 Stripperns dimensioner kommer också att bli stora, vilket innebär betydande kostnader för utrustningen.

I en vanlig ångstripper avdrivs även andra lättflyktiga komponenter, såsom metanol.

5 I stället för ånga kan man använda sig av luft för att strippa kondensatet. En stor nackdel med detta är att luften förorenas och att den måste renas på något sätt. Luftvolymerna kan också bli mycket stora. Dessutom kylv kondensatet ner av luften, som har en lägre våttemperatur än kondensatets temperatur. Därför är inte ren luftstrippning  
10 ett realistiskt alternativ för en modern och miljövänlig pappersmassafabrik.

Föreliggande uppfinning erbjuder en möjlighet att med mycket hög effektivitet driva av främst sulfider i lutång-kondensat  
15 från en cellulosaframställningsprocess, och att samtidigt ta hand om svavlet, så att det inte förorenar miljön. Detta sker genom att i en sluten krets bestående av tre processteg låta avdriva sulfiderna från kondensatet, oxidera de avdrivna sulfiderna till svaveldioxid och att absorbera denna  
20 svaveldioxid.

De tre processtegen blir således:

1. Avdrivning (strippning) av sulfider från lutång-kondensat.
2. Oxidering av brännbara komponenter som sulfider och  
25 kolväten.
3. Absorption av svaveldioxid.

Genom att låta dessa tre processteg (1, 2, 3) vara integrerade i en sluten krets kan själva reningen av  
30 kondensatet utföras med hög effektivitet och god energiekonomi, samtidigt som påverkan på miljön minimeras.

Uppfinningen kommer i det följande att exemplifieras med hänvisning till ett i bifogade ritning illustrerat schema,  
35 som schematiskt visar de olika processtegen enligt uppfinningen.



I föreliggande uppfinning användes en gas som medium för att driva av sulfiderna från kondensatet. Denna gas utgöres huvudsakligen och företrädesvis av luft. Detta processteg utformas normalt, som ett skrubbertorn 1, där gasen 4 tillföres i nedre delen och kondensatet 5 i övre delen så att gasen och kondensatet möts i motströmskontakt. Kontaktanordningar i skrubberna kan utgöras av bottnar eller packningsmaterial. Gasen 6 som lämnar skrubbern kommer att innehålla sulfider i form bl.a. av vätesulfid och metylmerkaptan, men även organiska komponenter, såsom metanol och terpener. Denna förorenade gas 6 förs till en oxidationsprocess 2 där gasen värmeväxlas motströms regenerativt. Avgaserna 7 från oxidationsprocessen innehåller till den viss del svaveldioxid. Dessa avgaser förs sedan till en kontaktanordning i form av en SO<sub>2</sub>-skrubber 3 där svaveldioxiden absorberas i en företrädesvis alkalisk lösning 8. Gasen förs sedan åter vidare till kondensats-skrubbern för att åter användas som avdrivningsmedium. På detta sätt bildas en sluten krets. Eftersom oxidationen i den slutna kretsen konsumerar syrgas måste nytt syre tillföras. Ytterligare syre kan tillsättas genom tillförsel 9 av företrädesvis luft eller annan syrehaltig gas. Systemet tillåter inte att gas ackumuleras i kretsen och därför måste en mindre mängd gas 10 blödas ut. Gascirkulationen drivs genom de tre stegen 25 företrädesvis av en fläkt.

Genom att gasen i den slutna kretsen huvudsakligen cirkulerar runt kan en förhöjning av olika gaskomponenter i systemet byggas upp till tämligen höga halter. Eftersom endast en mindre mängd gas blödes ut, blir emellertid utsläppen av för miljön skadliga komponenter mycket små trots höga koncentrationer i systemet.

Ett sätt att förbättra reningen av kondensatet i strippern är att låta öka SO<sub>2</sub>-koncentrationen efter SO<sub>2</sub>-skrubbern (3). Ett sådant förfarande kommer att medföra att kondensatet i strippern (1) får ett lägre pH-värde. Ett lägre pH-värde ger

i sin tur en bättre avdrivning av sulfider och möjliggör en närmast fullständig avdrivning av sulfider. En fullständig avdrivning kan annars vara svårt att erhålla eftersom kondensatet innehåller mindre mängder alkaliska komponenter som t.ex ammoniak som höjer kondensatets pH-värde när de sura sulfiderna avdrives. En alkalisk komponent som ammoniak kommer vid ett sänkt pH att stanna kvar i kondensatet. Därigenom undviks utsläpp av ammoniak, som annars efter oxidationen skulle omvandlas till kväveoxider.

10

En förhöjning av  $\text{SO}_2$ -halten efter  $\text{SO}_2$ -skrubbern (3) kan erhållas genom att justera tillförsel av alkali till detta steg så att absorptionsvätskans håller ett relativt lägre pH. Ju lägre pH desto högre  $\text{SO}_2$ -koncentration i gasen från skrubbersteget (3). Ju högre  $\text{SO}_2$ -halt i gasen, som utgör strippermediet, desto bättre avdrivning av sulfider från kondensatet. Denna effekt kan i sin tur användas på så sätt att kvoten mellan mängden kondensat och strippergas kan ökas med fortsatt god sulfidavdrivning. Detta i sin tur medför en förhöjning av halten avdrivna sulfider i strippergasen, vilket i sin tur innebär en förhöjd  $\text{SO}_2$ -halt efter oxidationen. På detta sätt kan  $\text{SO}_2$ -halten förhöjas avsevärt i hela systemet. Detta medför följande fördelar:

20

- 25 1. En produktion av sulfitsyra med ett relativt lågt pH möjliggörs.
2. Anläggningens storlek kan minskas.
3. Kväveutsläppet minskas (se ovan).

30

Den förnämnda fördelen möjliggörs genom att en förhöjd  $\text{SO}_2$ -halt i en gas jämviktsmässigt ger ett lägre pH i absorptionslösningen. Eftersom tillsats av alkali minskas kan en bisulfitsyra erhållas. Denna syra kan i sin tur nyttjas för surgörning i t.ex blekeriet eller tallolje-kokeriet. En förhöjning av  $\text{SO}_2$ -halten i den cirkulerande gasen medför dock att utsläppet av  $\text{SO}_2$  från systemet ökar via utblödningen till atmosfären (10). Detta kan dock åtgärdas genom att vid denna

35

punkt koppla in en skrubber, för att absorbera SO<sub>2</sub>. En skrubber i denna position utformas företrädesvis av flera absorptionssteg t.ex av samma utformning som strippern. Det skulle kunna förhålla sig så att man endast låter absorbera SO<sub>2</sub> i denna position. Således kan SO<sub>2</sub>-skrubbern (3) utslutas ur systemet.

Denna andra fördel följer av det förhållandet att anläggningens storlek huvudsakligen bestäms av det cirkulerande gasflödets storlek. Eftersom en förhöjd SO<sub>2</sub>-halt möjliggör att större kvoten mellan kondensat- och strippergasflöde, kommer gasflödet i systemet att kunna minskas.

Det renade kondensatet kommer att ha mycket låga sulfidhalter och även eventuella terpenier kommer att drivas av. Detta kommer att ge ett kondensat som är tämligen fritt från illaluktande föroreningar. En betydelsefull föroreningskomponent i lutångkondensat är metanol. En del av metanolen kommer att drivas av i strippern, en del kommer att finnas kvar i kondensatet. Andelen avdriven metanol kommer att vara beroende av förhållandet mellan tillförd mängd kondensat och mängden cirkulerande gas.

Värmeekonomin är god i systemet eftersom ingen extern värmeenergi behöver tillföras. I processen genereras dessutom energi i oxidationssteget. Denna energi kan kompensera olika energiförluster i systemet och eventuellt överskott kan tas upp som värme av det utgående kondensatet. I andra system där till exempel luft används som strippergas upptas betydande värmemängder i luften genom att det varma kondensatet avger vattenånga vid kontakten med luften. Detta ger en nedkylning av kondensatet, vilket undviks i den föreslagna uppfinningen, där eventuellt avdriven vattenånga återföres till systemet. Man kan eventuellt också utvinna värme ur systemet genom att låta införa en värmeväxlare i systemet. Med en sådan värmeväxlare som kyler systemet kan temperaturen styras.

Det kan även finnas anledning att tillföra värme till systemet. En anledning kan vara att man i vissa delar av kretsen vill undvika att gasen är fuktig. Eftersom den cirkulerande gasen efter t.ex. strippern är mättad med vattenånga finns risken att vattendroppar faller ut som fukt i gasen. Genom att värma gasen kan då fukten elimineras.

Investeringskostnader och storlek av komponenter står i stort sett i direkt proportion till mängden cirkulerande gas. Av denna anledning vill man begränsa den cirkulerande mängden gas. Detta kommer således att påverka metanol-avskiljningen. Det är därför rimligt att räkna med en viss mängd metanol som finns kvar i kondensatet. Metanol som förorening i kondensatet kan vara av nackdel om kondensatet skall släppas ut till recipienten. Om kondensatet däremot skall användas för internt bruk, som processvatten, t.ex. som tvättvatten i blekeri, massatvätt eller mesatvätt är ett sådant kondensat utmärkt trots metanolinnehållet.

Metanol har en positiv effekt på blekning, dels som radikalfångare och dels för förbättrad löslighet av lignin. Dessutom är detta kondensat metallfritt. Normalt processvatten som beretts från närliggande vattendrag innehåller alltid en del metaller, som bl.a. övergångsmetaller. Dessa övergångsmetaller kan vara mycket skadliga för blekeriprocessen eftersom dessa kan bryta ner blekerikemikalier, som t.ex. väteperoxid. Eftersom metanolen bidrar som radikalfångare, minskar nedbrytningen av blekerikemikalier. Ett metallfritt kondensat som används i blekeriet har därför stora fördelar trots ett visst metanolinnehåll. Genom återföring av kondensatet till processen undviks ett utsläpp av syreförbrukande ämnen. Metanolanrikningen i processen blir mycket marginell, eftersom uttaget av metanol från processen är relativt stor för varje processcykel.

Strippningen av kondensat kan utföras på en rad olika sätt. Den typ av utrustning, som bör väljas skall vara en

utrustning där avskiljningsgraden är mycket hög. En sådan  
utrustning bör bestå av ett flertal jämviktssteg efter  
varandra, där kondensatet möter en motgående gasström.  
Exempel på sådan utrustning är kolonner med bottnar eller  
5 packningsmaterial. Detta finns välk beskrivet i teknisk  
litteratur, som t.ex. "Perry's Chemical Engineers' Handbook",  
MacGraw-Hill Book Company, 1984.

10 Oxidationsprocessen kan ske på ett flertal sätt, men de  
relativt svaga koncentrationerna av brännbara föreningar  
ställer speciella krav på denna process. För att de brännbara  
föreningarna skall oxideras krävs en relativt hög  
temperatur. Man bör använda sig av en regenerativ termisk  
15 oxidationsprocess (RTO), där gasen värmväxlas så väl att  
erforderlig temperatur erhålles för en i det närmaste  
fullständig oxidation. Exempel på en sådan process beskrivs  
i patentansökan PCT/SE85/00257.

20 Skrubbing av de SO<sub>2</sub>-haltiga gaserna kan göras med en  
alkalisk vätska. Vid ett massabruk finns det god tillgång på  
alkaliska processvätskor. En sådan processvätska är oxiderad  
vitlut. I den oxiderade vitluten har sulfiden tagits bort  
genom oxidation. Vitlut är så starkt alkalisk att SO<sub>2</sub> mycket  
lätt kan absorberas. Det räcker utan vidare med ett  
25 jämviktssteg. En venturiskrubber är en processutrustning där  
knappt ett jämviktssteg erhålles. I venturiskrubbern kan en  
relativt hög gashastighet hållas, vilket gör utrustningen  
kompakt. Skrubbervätskan kommer att cirkuleras genom  
venturin.

30 Skrubbervätskans pH skall styras för att kontrollera SO<sub>2</sub>-  
halten i gaserna ut från skrubbern. Venturiskrubbern har en  
stor fördel genom att den cirkulerande vätskan kan ha  
relativt kort uppehållstid. Det möjliggör en snabb styrning  
35 av pH i skrubbern. Genom att skrubbern endast har ett knappt  
jämviktssteg i stället för flera blir svarstiden också snabb.

PATENTKRAV

1. Sätt att avlägsna sulfider och andra lättflyktiga  
5 föroreningar från lutångkondensat från en cellulosaframställningsprocess,  
k ä n n e t e c k n a t d ä r a v,  
att sagda lutångkondensat tillföres till en stripper (1), som  
ingår i en sluten krets bestående av sagda stripper (1) och  
10 en regenerativ termisk oxidations-process (RTO) (2), i vilken  
krets cirkuleras en gas (4) huvudsakligen bestående av luft  
och sådana komponenter, som bildas eller avdrivs i denna  
krets, varvid den cirkulerande gasen avdriver sulfider och  
andra lättflyktiga föroreningar från lutångkondensat (5),  
15 varefter gasströmmen (6) förs vidare till en RTO-process (2),  
där de avdrivna föroreningarna förbränns under bildande av  
SO<sub>2</sub> och därefter föres den SO<sub>2</sub>-haltiga gasen (7) till en SO<sub>2</sub>-  
skrubber (3), där företrädesvis alkali används som  
absorptionsmedel (8), varpå den cirkulerande gasen återföres  
20 till strippern (1).

2. Sätt enligt patentkravet 1,  
k ä n n e t e c k n a t d ä r a v,  
att SO<sub>2</sub>-skrubbern (3) ingår i den slutna kretsen.

25 3. Sätt enligt patentkravet 1 eller 2,  
k ä n n e t e c k n a t d ä r a v,  
att en mindre mängd gas (10) blödes ut ur kretsen samtidigt  
som luft eller annan syrehaltig gas (9) tillföres för att  
30 säkerställa att tillräckligt syre finns närvarande för att  
oxidationen i RTO-processen (2) skall ske.

4. Sätt enligt något av föregående patentkrav,  
k ä n n e t e c k n a t d ä r a v,  
35 att den som absorptionsmedel använda alkalien (8) är oxiderad  
vitlut.

5. Sätt enligt något av föregående patentkrav,  
k ä n n e t e c k n a t   d ä r a v,  
att surhetsgraden i SO<sub>2</sub>-skrubbern (3) regleras så att  
tillräcklig halt av SO<sub>2</sub> är kvar i gasen (4) när den återföres  
5 till strippen (1), där SO<sub>2</sub> försurar kondensatet (5) och  
därmed bidrar till att ytterligare avdriva sulfiderna i  
kondensatet.

10 6. Sätt enligt patentkravet 1,  
k ä n n e t e c k n a t   d ä r a v,  
att en värmeväxlare införes på godtycklig plats i den slutna  
kretsen för att utvinna eller tillföra energi och att  
därigenom styra temperaturen i systemet.

15 7. Sätt enligt patentkravet 1,  
k ä n n e t e c k n a t   d ä r a v,  
att man styr mängden recirkulerad gas gentemot mängden  
kondensat för optimering av metanolinnehållet i kondensatet.

20 8. Sätt enligt patentkravet 7,  
k ä n n e t e c k n a t   d ä r a v,  
att sagda kondensat användes som bakvatten i ett blekeri för  
att minska blekerikemikaliekostnaden.

25 9. Sätt enligt patentkravet 1,  
k ä n n e t e c k n a t   d ä r a v,  
att den gas (10) som blödes ut minimeras genom användning av  
ren syrgas eller en syrgasberikad luft, som för oxidationen  
nödvändig makeup-gas (9).

30 10. Sätt enligt patentkravet 1 eller 2,  
k ä n n e t e c k n a t   d ä r a v,  
att den utblöda gasen (10) från systemet skrubbas med  
avseende på SO<sub>2</sub> i en separat skrubber, som företrädesvis  
35 utgörs av ett flertal absorptionssteg.

11. Sätt enligt något av föregående patentkrav,  
k ä n n e t e c k n a t   d ä r a v ,  
att SO<sub>2</sub>-halten förhöjs till en sådan nivå i systemet att  
absorptionsvätskan i SO<sub>2</sub>-skrubbern blir tillräckligt sur för  
5 att denna vätska skall kunna nyttiggöras som surgörnings-  
medel i andra processdelar av en massafabriken som t.ex  
blekeri eller talloljekokeri.



SAMMANDRAG

Sätt att avlägsna sulfider och andra lättflyktiga  
föroreningar från lutångkondensat från en cellulosaför-  
5 framställningsprocess, varvid sagda lutkondensat tillföres  
till en stripper, som ingår i en sluten krets omfattande  
sagda stripper, en regenerativ termisk oxidations-process  
(RTO) och en SO<sub>2</sub>-skrubber, i vilken krets cirkuleras en gas  
huvudsakligen bestående av luft och sådana komponenter, som  
10 bildas eller avdrivs i denna krets, varvid den cirkulerande  
gasen avdriver sulfider och andra lättflyktiga föroreningar  
från lutångkondensat, varefter gasströmmen förs vidare till  
en RTO-process, där de avdrivna föroreningarna förbränns  
under bildande av SO<sub>2</sub> och därefter föres den SO<sub>2</sub>-haltiga  
15 gasen till en SO<sub>2</sub>-skrubber, där företrädesvis alkali används  
som absorptionsmedel, och därefter återföres den cirkulerande  
gasen till strippern.

(Fig. 1)

2000-08-25

From the INTERNATIONAL BUREAU

PCT

Göteborgs Patentbyrå Dahls AB

INFORMATION CONCERNING ELECTED  
OFFICES NOTIFIED OF THEIR ELECTION

(PCT Rule 61.3)

GÖTEBORGS PATENTBYRÅ DAHLS AB  
Sjöporten 4  
S-417 64 Göteborg  
SUÈDE

Date of mailing (day/month/year)

15 August 2000 (15.08.00)

Applicant's or agent's file reference

P15634PC/SC

## IMPORTANT INFORMATION

International application No.

PCT/SE99/02170

International filing date (day/month/year)

24 November 1999 (24.11.99)

Priority date (day/month/year)

26 November 1998 (26.11.98)

Applicant

EXCELENTEC HOLDING AB et al

1. The applicant is hereby informed that the International Bureau has, according to Article 31(7), notified each of the following Offices of its election:

AP : GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW

EP : AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE

National : AU, BG, BR, CA, CN, CZ, DE, IL, JP, KP, KR, MN, NO, NZ, PL, RO, RU, SE, SK, US

2. The following Offices have waived the requirement for the notification of their election; the notification will be sent to them by the International Bureau only upon their request:

EA : AM, AZ, BY, KG, KZ, MD, RU, TJ, TM

OA : BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG

National : AE, AL, AM, AT, AZ, BA, BB, BY, CH, CR, CU, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IN, IS, KE, KG, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MW, MX, PT, SD, SG, SI, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW

3. The applicant is reminded that he must enter the "national phase" before the expiration of 30 months from the priority date before each of the Offices listed above. This must be done by paying the national fee(s) and furnishing, if prescribed, a translation of the international application (Article 39(1)(a)), as well as, where applicable, by furnishing a translation of any annexes of the international preliminary examination report (Article 36(3)(b) and Rule 74.1).

Some offices have fixed time limits expiring later than the above-mentioned time limit. For detailed information about the applicable time limits and the acts to be performed upon entry into the national phase before a particular Office, see Volume II of the PCT Applicant's Guide.

The entry into the European regional phase is postponed until 31 months from the priority date for all States designated for the purposes of obtaining a European patent.

The International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland

Authorized officer:

F. Baechli

Facsimile No. (41-22) 740.14.36

Telephone No. (41-22) 338.83.38

3486123

COPY

From the INTERNATIONAL BUREAU

PCT

NOTICE INFORMING THE APPLICANT OF THE  
COMMUNICATION OF THE INTERNATIONAL  
APPLICATION TO THE DESIGNATED OFFICES

(PCT Rule 47.1(c), first sentence)

To:

GÖTEBORGS PATENTBYRÅ DAHLS AB  
Sjöporten 4  
S-417 64 Göteborg  
SUÈDE

2000-06-26 R

Date of mailing (day/month/year)  
15 June 2000 (15.06.00)Applicant's or agent's file reference  
P15634PC/SC

## IMPORTANT NOTICE

International application No.  
PCT/SE99/02170International filing date (day/month/year)  
24 November 1999 (24.11.99)Priority date (day/month/year)  
26 November 1998 (26.11.98)Applicant  
EXCELENTEC HOLDING AB et al

1. Notice is hereby given that the International Bureau has communicated, as provided in Article 20, the international application to the following designated Offices on the date indicated above as the date of mailing of this Notice:  
AU,CN,JP,KP,KR,US

In accordance with Rule 47.1(c), third sentence, those Offices will accept the present Notice as conclusive evidence that the communication of the international application has duly taken place on the date of mailing indicated above and no copy of the international application is required to be furnished by the applicant to the designated Office(s).

2. The following designated Offices have waived the requirement for such a communication at this time:

AE,AL,AM,AP,AT,AZ,BA,BB,BG,BR,BY,CA,CH,CR,CU,CZ,DE,DK,DM,EA,EE,EP,ES,FI,GB,GD,GE,  
GH,GM,HR,HU,ID,IL,IN,IS,KE,KG,KZ,LC,LK,LR,LS,LT,LU,LV,MA,MD,MG,MK,MN,MW,MX,NO,NZ,  
OA,PL,PT,RO,RU,SD,SE,SG,SI,SK,SL,TJ,TM,TR,TT,TZ,UA,UG,UZ,VN,YU,ZA,ZW

The communication will be made to those Offices only upon their request. Furthermore, those Offices do not require the applicant to furnish a copy of the international application (Rule 49.1(a-bis)).

3. Enclosed with this Notice is a copy of the international application as published by the International Bureau on  
15 June 2000 (15.06.00) under No. WO 00/34569

## REMINDER REGARDING CHAPTER II (Article 31(2)(a) and Rule 54.2)

If the applicant wishes to postpone entry into the national phase until 30 months (or later in some Offices) from the priority date, a demand for international preliminary examination must be filed with the competent International Preliminary Examining Authority before the expiration of 19 months from the priority date.

It is the applicant's sole responsibility to monitor the 19-month time limit.

Note that only an applicant who is a national or resident of a PCT Contracting State which is bound by Chapter II has the right to file a demand for international preliminary examination.

## REMINDER REGARDING ENTRY INTO THE NATIONAL PHASE (Article 22 or 39(1))

If the applicant wishes to proceed with the international application in the national phase, he must, within 20 months or 30 months, or later in some Offices, perform the acts referred to therein before each designated or elected Office.

For further important information on the time limits and acts to be performed for entering the national phase, see the Annex to Form PCT/IB/301 (Notification of Receipt of Record Copy) and Volume II of the PCT Applicant's Guide.

The International Bureau of WIPO  
34, chemin des Colombettes  
1211 Geneva 20, Switzerland

Authorized officer -

J. Zahra

Telephone No. (41-22) 338.83.38

Facsimile No. (41-22) 740.14.35

3338168

COPY

PCT

## REQUEST

The undersigned requests that the present international application be processed according to the Patent Cooperation Treaty.

For receiving Office use only

International Application No.

International Filing Date

Name of receiving Office and "PCT International Application"

Applicant's or agent's file reference  
(if desired) (12 characters maximum)

P15634PC/SC

Box No. I TITLE OF INVENTION A method for cleaning sulphide-containing condensates

## Box No. II APPLICANT

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)

EXELENTEC AB  
Bror Nilssons gata 16  
S-417 55 GÖTEBORG  
Sweden

☐ This person is also inventor.

Telephone No.

Facsimile No.

Teleprinter No.

State (i.e. country) of nationality: Sweden

State (that is, country) of residence: Sweden

This person is applicant  
for the purposes of:☐ all designated  
States☒ all designated States except  
the United States of America☐ the United States  
of America only☐ the States indicated in  
the Supplemental Box

## Box No. III FURTHER APPLICANT(S) AND/OR (FURTHER) INVENTOR(S)

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (i.e. country) of residence if no State of residence is indicated below.)

SANDQUIST, KENT K  
Askims Fornborgsv. 2  
S-436 42 ASKIM  
Sweden

This person is:

☐ applicant only☒ applicant and inventor☐ inventor only (If this check-box  
is marked, do not fill in below.)

State (that is, country) of nationality: Sweden

State (that is, country) of residence: Sweden

This person is applicant  
for the purposes of:☐ all designated  
States☐ all designated States except  
the United States of America☒ the United States  
of America only☐ the States indicated in  
the Supplemental Box☒ Further applicants and/or (further) inventors are indicated on a continuation sheet.

## Box No. IV AGENT OR COMMON REPRESENTATIVE: OR ADDRESS FOR CORRESPONDENCE

The person identified below is hereby/has been appointed to act on behalf  
of the applicant(s) before the competent International Authorities as:

☒ agent☐ common representative

Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)

GÖTEBORGS PATENTBYRÅ DAHLS AB  
Sjöporten 4  
S-417 64 GÖTEBORG  
Sweden

Telephone No.

+46-31-507700

Facsimile No.

+46-31-7790540

Teleprinter No.

☐ Address for correspondence: Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

Continuation of Box No. III <b>FURTHER APPLICANTS AND/OR (FURTHER) INVENTORS</b>	
<i>If none of the following sub-boxes is used, this sheet is not to be included in the request.</i>	
<p>Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</p> <p>WENNBERG, Olle Vaktmästaregängen 1 S-413 18 GÖTEBORG Sweden</p>	<p>This person is:</p> <p><input type="checkbox"/> applicant only</p> <p><input checked="" type="checkbox"/> applicant and inventor</p> <p><input type="checkbox"/> inventor only (if this check-box is marked, do not fill in below.)</p>
<p>State (that is, country) of nationality: Sweden</p>	<p>State (that is, country) of residence: Sweden</p>
<p>This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input checked="" type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box</p>	
<p>Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</p>	<p>This person is:</p> <p><input type="checkbox"/> applicant only</p> <p><input type="checkbox"/> applicant and inventor</p> <p><input type="checkbox"/> inventor only (if this check-box is marked, do not fill in below.)</p>
<p>State (that is, country) of nationality:</p>	<p>State (that is, country) of residence:</p>
<p>This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box</p>	
<p>Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</p>	<p>This person is:</p> <p><input type="checkbox"/> applicant only</p> <p><input type="checkbox"/> applicant and inventor</p> <p><input type="checkbox"/> inventor only (if this check-box is marked, do not fill in below.)</p>
<p>State (that is, country) of nationality:</p>	<p>State (that is, country) of residence:</p>
<p>This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box</p>	
<p>Name and address: (Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country. The country of the address indicated in this Box is the applicant's State (that is, country) of residence if no State of residence is indicated below.)</p>	<p>This person is:</p> <p><input type="checkbox"/> applicant only</p> <p><input type="checkbox"/> applicant and inventor</p> <p><input type="checkbox"/> inventor only (if this check-box is marked, do not fill in below.)</p>
<p>State (that is, country) of nationality:</p>	<p>State (that is, country) of residence:</p>
<p>This person is applicant for the purposes of: <input type="checkbox"/> all designated States <input type="checkbox"/> all designated States except the United States of America <input type="checkbox"/> the United States of America only <input type="checkbox"/> the States indicated in the Supplemental Box</p>	
<p><input type="checkbox"/> Further applicants and/or (further) inventors are indicated on another continuation sheet.</p>	

Box No. V DESIGNATION OF STATES

The following designations are hereby made under Rule 4.9(a) (mark the applicable check-boxes: at least one must be marked):

Regional Patent

- ☒ **AP** ARIPO Patent: GH Ghana, GM Gambia, KE Kenya, LS Lesotho, MW Malawi, SD Sudan, SZ Swaziland, UG Uganda, ZW Zimbabwe and any other State which is a Contracting State of the Harare Protocol and of the PCT
- ☒ **EA** Eurasian Patent: AM Armenia, AZ Azerbaijan, BY Belarus, KG Kyrgyzstan, KZ Kazakhstan, MD Republic of Moldova, RU Russian Federation, TJ Tajikistan, TM Turkmenistan, and any other State which is a Contracting State of the Eurasian Patent Convention and of the PCT
- ☒ **EP** European Patent: AT Austria, BE Belgium, CH and LI Switzerland and Liechtenstein, CY Cyprus, DE Germany, DK Denmark, ES Spain, FI Finland, FR France, GB United Kingdom, GR Greece, IE Ireland, IT Italy, LU Luxembourg, MC Monaco, NL Netherlands, PT Portugal, SE Sweden, and any other State which is a Contracting State of the European Patent Convention and of the PCT
- ☒ **OA** OAPI Patent: BF Burkina Faso, BJ Benin, CF Central African Republic, CG Congo, CI Côte d'Ivoire, CM Cameroon, GA Gabon, GN Guinea, GW Guinea-Bissau, ML Mali, MR Mauritania, NE Niger, SN Senegal, TD Chad, TG Togo, and any other State which is a member State of OAPI and a Contracting State of PCT (if other kind of protection or treatment desired, specify on dotted line) -

National Patent (if other kind of protection or treatment desired specify on dotted line)

- |   |   |
|---|---|
| <input checked="" type="checkbox"/> <b>AE</b> United Arab Emirates                  | <input checked="" type="checkbox"/> <b>LR</b> Liberia                                   |
| <input checked="" type="checkbox"/> <b>AL</b> Albania                               | <input checked="" type="checkbox"/> <b>LS</b> Lesotho                                   |
| <input checked="" type="checkbox"/> <b>AM</b> Armenia                               | <input checked="" type="checkbox"/> <b>LT</b> Lithuania                                 |
| <input checked="" type="checkbox"/> <b>AT</b> Austria                               | <input checked="" type="checkbox"/> <b>LU</b> Luxembourg                                |
| <input checked="" type="checkbox"/> <b>AU</b> Australia                             | <input checked="" type="checkbox"/> <b>LV</b> Latvia                                    |
| <input checked="" type="checkbox"/> <b>AZ</b> Azerbaijan                            | <input checked="" type="checkbox"/> <b>MD</b> Republic of Moldova                       |
| <input checked="" type="checkbox"/> <b>BA</b> Bosnia and Herzegovina                | <input checked="" type="checkbox"/> <b>MG</b> Madagascar                                |
| <input checked="" type="checkbox"/> <b>BB</b> Barbados                              | <input checked="" type="checkbox"/> <b>MK</b> The former Yugoslav Republic of Macedonia |
| <input checked="" type="checkbox"/> <b>BG</b> Bulgaria                              | <input checked="" type="checkbox"/> <b>MN</b> Mongolia                                  |
| <input checked="" type="checkbox"/> <b>BR</b> Brazil                                | <input checked="" type="checkbox"/> <b>MW</b> Malawi                                    |
| <input checked="" type="checkbox"/> <b>BY</b> Belarus                               | <input checked="" type="checkbox"/> <b>MX</b> Mexico                                    |
| <input checked="" type="checkbox"/> <b>CA</b> Canada                                | <input checked="" type="checkbox"/> <b>NO</b> Norway                                    |
| <input checked="" type="checkbox"/> <b>CH and LI</b> Switzerland and Liechtenstein  | <input checked="" type="checkbox"/> <b>NZ</b> New Zealand                               |
| <input checked="" type="checkbox"/> <b>CN</b> China                                 | <input checked="" type="checkbox"/> <b>PL</b> Poland                                    |
| <input checked="" type="checkbox"/> <b>CR</b> Costa Rica                            | <input checked="" type="checkbox"/> <b>PT</b> Portugal                                  |
| <input checked="" type="checkbox"/> <b>CU</b> Cuba                                  | <input checked="" type="checkbox"/> <b>RO</b> Romania                                   |
| <input checked="" type="checkbox"/> <b>CZ</b> Czech Republic                        | <input checked="" type="checkbox"/> <b>RU</b> Russian Federation                        |
| <input checked="" type="checkbox"/> <b>DE</b> Germany                               | <input checked="" type="checkbox"/> <b>SD</b> Sudan                                     |
| <input checked="" type="checkbox"/> <b>DK</b> Denmark                               | <input checked="" type="checkbox"/> <b>SE</b> Sweden                                    |
| <input checked="" type="checkbox"/> <b>DM</b> Dominica                              | <input checked="" type="checkbox"/> <b>SG</b> Singapore                                 |
| <input checked="" type="checkbox"/> <b>EE</b> Estonia                               | <input checked="" type="checkbox"/> <b>SI</b> Slovenia                                  |
| <input checked="" type="checkbox"/> <b>ES</b> Spain                                 | <input checked="" type="checkbox"/> <b>SL</b> Sierra Leone                              |
| <input checked="" type="checkbox"/> <b>FI</b> Finland                               | <input checked="" type="checkbox"/> <b>SK</b> Slovakia                                  |
| <input checked="" type="checkbox"/> <b>GB</b> United Kingdom                        | <input checked="" type="checkbox"/> <b>TJ</b> Tajikistan                                |
| <input checked="" type="checkbox"/> <b>GD</b> Grenada                               | <input checked="" type="checkbox"/> <b>TM</b> Turkmenistan                              |
| <input checked="" type="checkbox"/> <b>GE</b> Georgia                               | <input checked="" type="checkbox"/> <b>TR</b> Turkey                                    |
| <input checked="" type="checkbox"/> <b>GH</b> Ghana                                 | <input checked="" type="checkbox"/> <b>TT</b> Trinidad and Tobago                       |
| <input checked="" type="checkbox"/> <b>GM</b> Gambia                                | <input checked="" type="checkbox"/> <b>TZ</b> United Republic of Tanzania               |
| <input checked="" type="checkbox"/> <b>HR</b> Croatia                               | <input checked="" type="checkbox"/> <b>UA</b> Ukraine                                   |
| <input checked="" type="checkbox"/> <b>HU</b> Hungary                               | <input checked="" type="checkbox"/> <b>UG</b> Uganda                                    |
| <input checked="" type="checkbox"/> <b>ID</b> Indonesia                             | <input checked="" type="checkbox"/> <b>US</b> United States of America                  |
| <input checked="" type="checkbox"/> <b>IL</b> Israel                                | <input checked="" type="checkbox"/> <b>UZ</b> Uzbekistan                                |
| <input checked="" type="checkbox"/> <b>IN</b> India                                 | <input checked="" type="checkbox"/> <b>VN</b> Viet Nam                                  |
| <input checked="" type="checkbox"/> <b>IS</b> Iceland                               | <input checked="" type="checkbox"/> <b>YU</b> Yugoslavia                                |
| <input checked="" type="checkbox"/> <b>JP</b> Japan                                 | <input checked="" type="checkbox"/> <b>ZA</b> South Africa                              |
| <input checked="" type="checkbox"/> <b>KE</b> Kenya                                 | <input checked="" type="checkbox"/> <b>ZW</b> Zimbabwe                                  |
| <input checked="" type="checkbox"/> <b>KG</b> Kyrgyzstan                            |   |
| <input checked="" type="checkbox"/> <b>KP</b> Democratic People's Republic of Korea |   |
| <input checked="" type="checkbox"/> <b>KR</b> Republic of Korea                     |   |
| <input checked="" type="checkbox"/> <b>KZ</b> Kazakhstan                            |   |
| <input checked="" type="checkbox"/> <b>LC</b> Saint Lucia                           |   |
| <input checked="" type="checkbox"/> <b>LK</b> Sri Lanka                             |   |

Check-boxes reserved for designating States (for the purposes of a national patent) which have become party to the PCT after issuance of this sheet:

☒ **MA** Morocco

**Precautionary Designation Statement:** In addition to the designations made above, the applicant also makes under Rule 4.9(b) all other designations which would be permitted under the PCT except any designation(s) indicated in the Supplemental Box as being excluded from the scope of this statement. The applicant declares that those additional designations are subject to confirmation and that any designation which is not confirmed before the expiration of 15 months from the priority date is to be regarded as withdrawn by the applicant at the expiration of that time limit. Confirmation of a designation consists of the filing of a notice specifying that designation and the payment of the designation and confirmation fees. Confirmation must reach the receiving Office within the 15-month time limit.

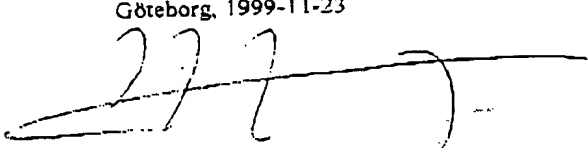
<input type="checkbox"/> Further priority claims are indicated in the Supplemental Box.				
<b>Box No. VI PRIORITY CLAIM</b>				
Filing date of earlier application (day/month/year)	Number of earlier application	Where earlier application is:		
		national application: country	regional application: regional Office	international application: receiving Office
item (1) 26.11.98	9804061-1	Sweden		
item (2)				
item (3)				

☒ The receiving Office is requested to prepare and transmit to the International Bureau a certified copy of the earlier application(s) (only if the earlier application was filed with the Office which for the purposes of the present international application is the receiving Office) identified above as item(s): **SE 9804061-1**

\* Where the earlier application is an ARIPO application, it is mandatory to indicate in the Supplemental Box at least one country party to the Paris Convention for the Protection of Industrial Property for which that earlier application was filed (Rule 4.10(b)(ii)). See Supplemental Box.

<b>Box No. VII INTERNATIONAL SEARCHING AUTHORITY</b>		
Choice of International Searching Authority (ISA) (if two or more International Searching Authorities are competent to carry out the international search, indicate the Authority chosen; the two-letter code may be used):	Request to use results of earlier search; reference to that search (if an earlier search has been carried out by or requested from the International Searching Authority):	
ISA /	Date (day/month/year)	Number                      Country (or regional Office)

<b>Box No. VIII CHECK LIST; LANGUAGE OF FILING.</b>	
This international application contains the following number of sheets:  request : 4  description (excluding sequencelisting part) : 8  claims : 3  abstract : 1  drawings : 1  sequence listing part of description : _____  Total number of sheets : 17	This international application is accompanied by the item(s) marked below: 1. <input checked="" type="checkbox"/> fee calculation sheet 2. <input checked="" type="checkbox"/> separate signed power of attorney 3. <input type="checkbox"/> copy of general power of attorney; reference number, if any: 4. <input type="checkbox"/> statement explaining lack of signature 5. <input type="checkbox"/> priority document(s) identified in Box No. VI as item(s): 6. <input type="checkbox"/> translation of international application into (language): 7. <input type="checkbox"/> separate indications concerning deposited microorganism or other biological material 8. <input type="checkbox"/> nucleotide and/or amino acid sequence listing in computer readable form 9. <input type="checkbox"/> other (specify):
Figure of the drawings which should accompany the abstract: 1	Language of filing of the international application: <b>Swedish</b>

<b>Box No. IX SIGNATURE OF APPLICANT OR AGENT</b>	
Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the request).	
Göteborg, 1999-11-23    Börje Westman GÖTEBORGS PATENTBYRÅ DAHLS AB	

For receiving Office use only		2. Drawings:  <input type="checkbox"/> received:  <input type="checkbox"/> not received:
1. Date of actual receipt of the purported international application:		
3. Corrected date of actual receipt due to later but timely received papers or drawings completing the purported international application:		
4. Date of timely receipt of the required corrections under PCT Article 11(2):		
5. International Searching Authority (if two or more are competent): <b>ISA /</b>		
6. <input type="checkbox"/> Transmittal of search copy delayed until search fee is paid.		
For International Bureau use only		
Date of receipt of the record copy by the International Bureau:		

This sheet is not part and does not count as a sheet of the international application

**PCT**

**FEE CALCULATION SHEET**

Annex to the Request

For receiving Office use only

International application No.

Date stamp of the receiving Office

Applicant's or agent's  
file reference **P15634PC/SC**

Applicant **Exelentec AB, et.al.**

**CALCULATION OF PRESCRIBED FEES**

1. TRANSMITTAL FEE . . . . .

1000

T

2. SEARCH FEE . . . . .

8510

S

International search to be carried out by **SE**  
(If two or more International Searching Authorities are competent in relation to the international application, indicate the name of the Authority which is chosen to carry out the international search)

3. INTERNATIONAL FEE

**Basic Fee**

The international application contains **17** sheets

3500

b1

first 30 sheets . . . . .

0

b2

remaining sheets  $\times$  additional amount =

3500

B

Add amounts entered at b1 and b2 and enter total at B . . . .

**Designation Fees**

The international application contains \_\_\_\_\_ designations.

10

800

8000

D

number of designation fees  $\times$  amount of designation fee  
payable (maximum 10)

11500

I

Add amounts entered at B and D and enter total at I . . . . .

(Applicants from certain States are entitled to a reduction of 75% of the international fee. Where the applicants is /or all applicants are) so entitled, the total to be entered at I is 25% of the sum of the amounts entered at B and D.)

4. FEE FOR PRIORITY DOCUMENT (if applicable) . . . . .

P

5. TOTAL FEES PAYABLE . . . . .

21010

TOTAL

Add amounts entered at T, S, I and P, and enter total in the TOTAL box

☐ The designation fees are not paid at this time

**MODE OF PAYMENT**

☐ authorization to charge  
deposit account (see below)

☐ bank draft

☐ coupons

☒ cheque

☐ cash

☐ other (specify):

☐ postal money order

☐ revenue stamps

**DEPOSIT ACCOUNT AUTHORIZATION** (this mode of payment may not be available at all receiving Offices)

The RO/ \_\_\_\_\_ ☐ is hereby authorized to charge the total fees indicated above to my deposit account.

☐ (this check-box may be marked only if the conditions for deposit-accounts of the receiving Office so permit) is hereby authorized to charge any deficiency or credit any overpayment in the total fees indicated above to my deposit account

☐ is hereby authorized to charge the fee for preparation and transmittal of the priority document to the International Bureau of WIPO to my deposit account.

Deposit Account No.

Date (day/month/year)

Signature



# COPY

From the  
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To:

GÖTEBORGS PATENTBYRA DAHLS AB  
Sjöporten 4  
S-417 64 Göteborg  
SUEDE

PCT

NOTIFICATION OF TRANSMITTAL OF  
THE INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT  
(PCT Rule 71.1)

Date of mailing  
(day/month/year) 08.01.2001

Applicant's or agent's file reference  
P15634PC/SC

IMPORTANT NOTIFICATION

International application No.  
PCT/SE99/02170

International filing date (day/month/year)  
24/11/1999

Priority date (day/month/year)  
26/11/1998

Applicant  
EXCELENTEC HOLDING AB et al.

1. The applicant is hereby notified that this International Preliminary Examining Authority transmits herewith the international preliminary examination report and its annexes, if any, established on the international application.
2. A copy of the report and its annexes, if any, is being transmitted to the International Bureau for communication to all the elected Offices.
3. Where required by any of the elected Offices, the International Bureau will prepare an English translation of the report (but not of any annexes) and will transmit such translation to those Offices.

4. REMINDER

The applicant must enter the national phase before each elected Office by performing certain acts (filing translations and paying national fees) within 30 months from the priority date (or later in some Offices) (Article 39(1)) (see also the reminder sent by the International Bureau with Form PCT/IB/301).

Where a translation of the international application must be furnished to an elected Office, that translation must contain a translation of any annexes to the international preliminary examination report. It is the applicant's responsibility to prepare and furnish such translation directly to each elected Office concerned.

For further details on the applicable time limits and requirements of the elected Offices, see Volume II of the PCT Applicant's Guide.

Name and mailing address of the IPEA/

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## PCT

## INTERNATIONAL PRELIMINARY EXAMINATION REPORT

(PCT Article 36 and Rule 70)

Applicant's or agent's file reference <b>P15634PC/SC</b>		<b>FOR FURTHER ACTION</b> See Notification of Transmittal of International Preliminary Examination Report (Form PCT/IPEA/416)	
International application No. <b>PCT/SE99/02170</b>	International filing date (day/month/year) <b>24/11/1999</b>	Priority date (day/month/year) <b>26/11/1998</b>	
International Patent Classification (IPC) or national classification and IPC <b>D21C11/08</b>			
Applicant <b>EXCELENTEC HOLDING AB et al.</b>			
<p>1. This international preliminary examination report has been prepared by this International Preliminary Examining Authority and is transmitted to the applicant according to Article 36.</p> <p>2. This REPORT consists of a total of 4 sheets, including this cover sheet.</p> <p><input type="checkbox"/> This report is also accompanied by ANNEXES, i.e. sheets of the description, claims and/or drawings which have been amended and are the basis for this report and/or sheets containing rectifications made before this Authority (see Rule 70.16 and Section 607 of the Administrative Instructions under the PCT).</p> <p>These annexes consist of a total of sheets.</p>			
<p>3. This report contains indications relating to the following items:</p> <p>I <input checked="" type="checkbox"/> Basis of the report</p> <p>II <input type="checkbox"/> Priority</p> <p>III <input type="checkbox"/> Non-establishment of opinion with regard to novelty, inventive step and industrial applicability</p> <p>IV <input type="checkbox"/> Lack of unity of invention</p> <p>V <input checked="" type="checkbox"/> Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement</p> <p>VI <input type="checkbox"/> Certain documents cited</p> <p>VII <input type="checkbox"/> Certain defects in the international application</p> <p>VIII <input checked="" type="checkbox"/> Certain observations on the international application</p>			
Date of submission of the demand  <b>13/06/2000</b>		Date of completion of this report  <b>08.01.2001</b>	
Name and mailing address of the international preliminary examining authority:  <b>European Patent Office</b> <b>D-80298 Munich</b> Tel. +49 89 2339 - 0 Tx: 523656 epmu d Fax: +49 89 2339 - 4465		Authorized officer <b>Tragoustis, M</b> Telephone No. +49 89 2339 8623 	

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/SE99/02170

**I. Basis of the report**

1. This report has been drawn on the basis of *(substitute sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this report as "originally filed" and are not annexed to the report since they do not contain amendments (Rules 70.16 and 70.17).)*  
Description, pages:

1-8 as originally filed

**Claims, No.:**

1-11 as originally filed

**Drawings, sheets:**

1/1 as originally filed

2. With regard to the language, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.

These elements were available or furnished to this Authority in the following language: , which is:

- ☐ the language of a translation furnished for the purposes of the international search (under Rule 23.1(b)).
- ☐ the language of publication of the international application (under Rule 48.3(b)).
- ☐ the language of a translation furnished for the purposes of international preliminary examination (under Rule 55.2 and/or 55.3).

3. With regard to any nucleotide and/or amino acid sequence disclosed in the international application, the international preliminary examination was carried out on the basis of the sequence listing:

- ☐ contained in the international application in written form.
- ☐ filed together with the international application in computer readable form.
- ☐ furnished subsequently to this Authority in written form.
- ☐ furnished subsequently to this Authority in computer readable form.
- ☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
- ☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. The amendments have resulted in the cancellation of:

- ☐ the description, pages:
- ☐ the claims, Nos.:

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT**

International application No. PCT/SE99/02170

☐ the drawings, sheets:

5. ☐ This report has been established as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed (Rule 70.2(c)):

*(Any replacement sheet containing such amendments must be referred to under item 1 and annexed to this report.)*

6. Additional observations, if necessary:

**V. Reasoned statement under Article 35(2) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement**

**1. Statement**

Novelty (N)	Yes:	Claims	1-11
	No:	Claims	

Inventive step (IS)	Yes:	Claims	1-11
	No:	Claims	

Industrial applicability (IA)	Yes:	Claims	1-11
	No:	Claims	

**2. Citations and explanations  
see separate sheet**

**VIII. Certain observations on the international application**

The following observations on the clarity of the claims, description, and drawings or on the question whether the claims are fully supported by the description, are made:  
**see separate sheet**

**INTERNATIONAL PRELIMINARY  
EXAMINATION REPORT - SEPARATE SHEET**

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International application No. PCT/SE99/02170

1. The application relates to a method of removing sulphides and other volatile contaminants from liquor vapour condensate originating from a pulp manufacturing process.

According to claim 1 the sulphides are stripped off in a stripper from the condensate by a gas, which gas is then fed to a regenerative oxidation system, where the stripped gases are combusted and  $\text{SO}_2$  is formed. The gas containing the  $\text{SO}_2$  is then lead to a  $\text{SO}_2$  scrubber and the gas from the scrubber is recirculated to the stripper.

By this closed loop of the gas, the cleaning of the condensates is carried out with high efficiency and good heat economy.

The documents of the Search Report are silent about the above three stages (stripping, oxidation, scrubbing) integrated in a closed loop cycle of the stripping gas.

WO 86/00389 discloses a process for combustion of pollutants carried in a gas. Sulfide contaminated condensates as well as stripping are not mentioned.

GB-A-1354499 discloses a process, whereby organic sulphur compounds present in condensates are stripped by an oxygen stream and are then catalytically oxidised. A closed loop for the stripping gas is not mentioned.

WO 98/55685 deals with the removal of sulphur from non-condensable gas stream released during the Kraft process.

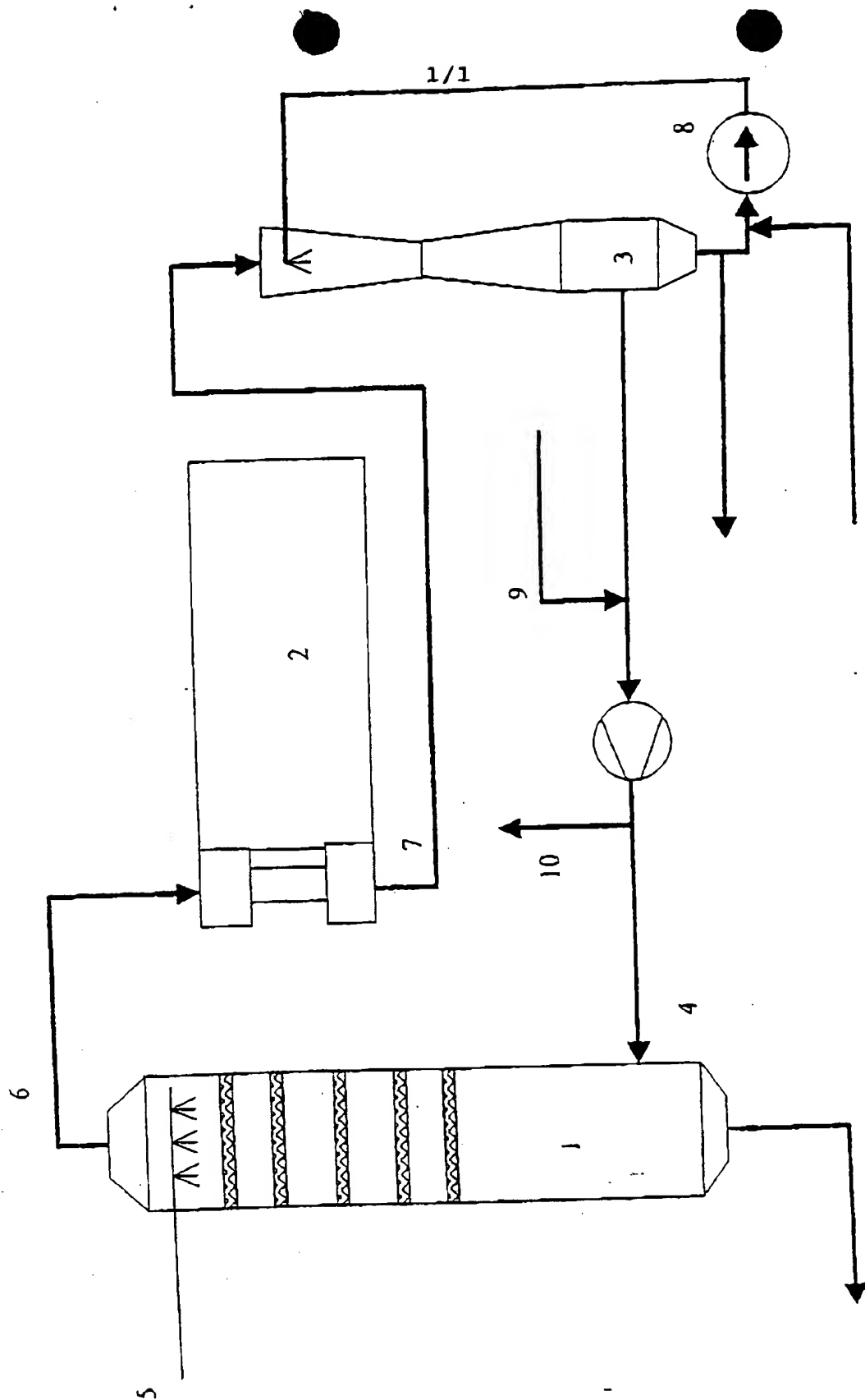
Hence claim 1 and the dependent claims 2-11 meet the requirements of Art. 33 PCT.

2. Apparently (see also page 3,4 of the description) the  $\text{SO}_2$  scrubber is part of the closed gas loop.

Hence the feature of claim 2 being an essential feature of the claimed invention should be included in claim 1 (Art. 6 PCT).

The word "either" in claim 1 causes unclarity and should be deleted.

FIG 1





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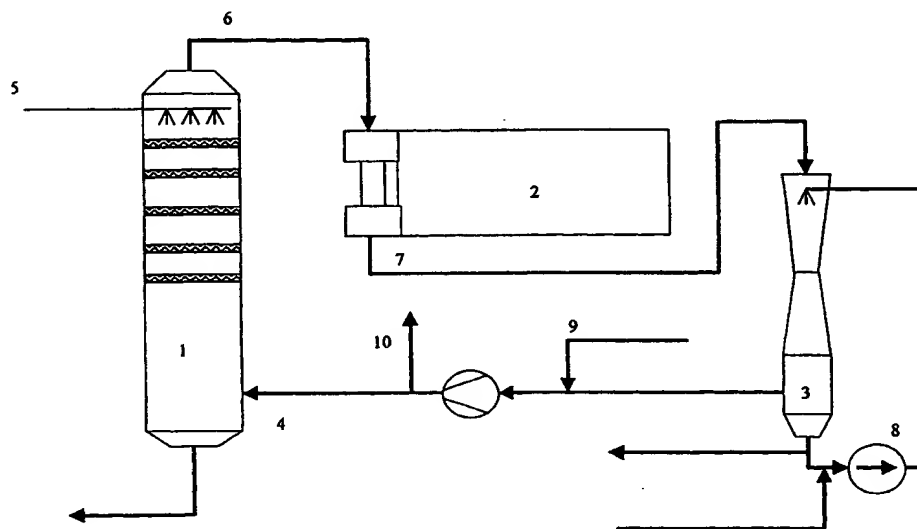
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(54) Title: A METHOD OF CLEANING SULFIDE CONTAMINATED CONDENSATES



## (57) Abstract

A method to remove sulphides and other volatile contaminants from liquor vapor condensate in a pulp manufacturing process, where the mentioned liquor vapor condensate is fed into a stripper, which is part of a closed loop system comprising said stripper, a regenerative thermal oxidization process (RTO) and a SO<sub>2</sub> scrubber, in which loop a gas is circulated, preferably air and such components formed or stripped off, in this loop whereafter the circulating gas is stripping off sulphides and other volatile components from the liquor vapor condensate, whereafter the gas stream is fed into an RTO process, where the stripped off contaminants are combusted and under formation of SO<sub>2</sub> and thereafter the SO<sub>2</sub> enriched gas is led to a SO<sub>2</sub> scrubber, where preferably alkali is used as absorption medium, and thereafter the circulating gas is returned back into the stripper.

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## **A method of cleaning sulfide contaminated condensates**

In producing chemical pulp according to the Kraft chemical pulp process, waste liquor is produced that is being evaporated prior to burning. During the evaporation process, liquor vapor is stripped off, which in addition to water vapor, also contains certain volatile contaminants. Such contaminants are hydrogen sulfide, methylmercaptan, dimethylsulfide, methanol, terpenes etc. At the evaporation which takes place as a so called multiple effect evaporation with a number of stages, effects (normally 4 - 7), the liquor vapor is also condensed in multiple stages, whereby also large amounts of the volatile contaminants will condense. The condensation takes place in at least as many stages there are effects. This means that the quality of the condensate varies significantly from the different stages of the evaporation. Normally 2-3 different condensate qualities are being separated, where each one is a mixture of condensates from a number of effects. The dirtiest condensate, (foul condensate), is normally treated in a steam stripper where the volatile components are flashed off. This foul condensate is typically a small amount of the total condensate flow and therefore the steam economy is not affected to any higher degree of the fact that steam is used as the stripper gas. The investment cost can also be kept at a minimum.

The purity of the other condensate qualities is highly dependent on the amount of foul condensate. If the amount of foul condensate is increased the contaminated condensates will be cleaner. A too high amount of foul condensate however the operating and investment cost for the steam stripper system will increase.

The other, less contaminated condensates can to a limited extent be used as process water in dependency of their cleanliness. However if the condensate is too contaminated it can not be re-used but must instead be discharged to the recipient subsequent to some form of treatment.

The primary limiting factor for the use of the contaminated condensate as process water is the content of sulfides, as these can give an unpleasant smell and taste to the pulp. It also creates a significant problem for the working environment. Also terpenes give a smell.

The terpenes however are normally present at very low amounts in the less contaminated condensates.

The technology available to clean these condensates is predominately steam stripping. Since the various condensate flows are very large, the size of the stripper will be significant and a large amount of steam will be required for stripping. The steam volumes will be so large that it will definitely not be economical to use fresh steam. On the other hand it is possible to use flash steam driven off from the evaporation of the waste liquor, in multiple effect evaporation for the stripping. The steam leaving the stripper then can be regained as heat in the next evaporation effect. The cleaning efficiency of such a stripper is however limited since the flash steam from the preceding effect is already contaminated with sulfides, which limits the degree of purity of the output condensate. Primarily the cleanliness is limited regarding sulphides, as the waste liquor can have a considerable content of sulphides. This sulphide content is dependent on that steam is normally taken from the first effect, where the temperature is rather high, which gives an increased sulphide content.

Another drawback is that when the steam passes through the stripper, it loses pressure and volatile components are enriched. These two things will reduce the condensation temperature, which means that the temperature difference available at the evaporation is reduced. The energy and capital cost are both negatively impacted thereby. Furthermore the evaporation plant and the stripper are completely integrated, whereby these two parts can not be independently operated.

The dimensions of the stripper also will become large, which means significant costs for the equipment.

In a conventional steam stripper also other volatile components, such as methanol, are stripped off.

Air can be used to in lieu of steam to strip the condensates. A big drawback with this method is that air is being contaminated and must be cleaned in some way. The air

volumes can also be very large. Additionally the condensate is being cooled down by the air, which has a lower wet bulb temperature as compared to the temperature of the condensate. For these reasons pure air stripping is not a realistic alternative for a modern and environmentally friendly pulp mill.

5

The present invention provides a possibility to strip off primarily sulfides at a very high efficiency from liquor-steam condensates from a pulp manufacturing process, and simultaneously to take care of the sulphur, thus that it will not contaminate the environment. This is being done in a closed loop concept that is comprised of three  
10 process steps, where the sulfides are stripped off from the condensate, the stripped off sulfides are being oxidized to sulphur dioxide, and to absorb the sulphur dioxide formed.

The three process steps are consequently:

1. Stripping off sulphides from liquor-steam condensate
- 15 2. Oxidation of combustible components such as sulphides and hydro carbons.
3. Absorption of sulphur dioxide.

By integrating these three process steps (1, 2, and 3) in a closed loop cycle, the cleaning of condensates can be done with a high efficiency, good heat economy, and minimal  
20 impact on the environment

The invention will in the following text be exemplified with reference to a scheme shown in the attached drawing, which schematically shows the various process steps in accordance with the invention.

25

In the present invention a gas is used as a medium for stripping off the sulphides from the condensate. This gas is substantially and preferably composed of air. This process step is normally designed as a scrubber column 1, where the gas 4 is introduced in the lower section and the condensate 5 in the upper section, thus that the gas and the condensate meet  
30 in counterflow contact. The contact means in the scrubber can be trays or packing material. The gas 6 leaving the scrubber will contain sulphides in form i.a. of hydrogen

5 sulphide and methyl mercaptan, but also organic compounds such as methanol and terpenes. This contaminated gas 6 is led to an oxidization process 2, where the gas is treated counterflow in a regenerative heat exchanger. The gas 7 from the oxidization step contains partly sulphur dioxide. These gases are then fed to a contact device, in form of a SO<sub>2</sub> scrubber 3, where the sulphur dioxide is absorbed in a preferably alkaline solution 8. The gas is then returned to the condensate scrubber to be used again as a stripping medium. In this manner is formed a closed the loop. Since oxidation in the closed loop consumes oxygen is necessary to add fresh oxygen. Additional oxygen can be added by supply 9 preferably of air or some other oxygen containing gas. The system does not allow for gas accumulation in the loop and therefore a minor portion of the gas 10 must be bled off. The gas circulation through the three process steps is accomplished by the use preferably of a fan.

15 Since the gas in the closed loop is primarily being circulated, an elevated level of various gas components can accumulate to rather high levels. However, since only a minor portion of the gas is bled off, the discharge of components harmful to the environment, will be limited, in spite of high concentrations in the system.

20 A method of improving the cleaning of the condensate in the stripper is to increase the level of SO<sub>2</sub> after the SO<sub>2</sub> scrubber (3). Such a method will result in that the condensate in the stripper (1) will get a lower pH value. A lower pH value in turn gives a better stripping of sulphides and makes possible an almost complete stripping of sulphides. This would otherwise be difficult to achieve since the condensate contains a smaller amount of alkali components, i.e. ammonia, which would increase the pH value of the condensate when the acidic sulfides are stripped off. An alkali component such as ammonia will remain in the 25 condensate at a lowered pH. Thereby is avoided discharge of ammonia, which should otherwise be transformed to Nox, after the oxidation process.

30 An increase of the SO<sub>2</sub> concentration after the SO<sub>2</sub> scrubber (3) can be obtained by adjusting the supply of alkali to this stage thus that the te absorption medium will get a

comparatively lower pH. The lower the pH the higher the SO<sub>2</sub> concentration in the gas leaving the scrubber (3). The higher the SO<sub>2</sub>-level in the gas, which constitutes the stripper media, the better the efficiency of stripping off sulfides from the condensate. In turn this effect can be utilized in such a way that the ratio between the condensate flow and stripper gas flow can be increased with continuous good sulphide stripping. This in turn implies an elevated level of sulphides in the stripper off gases, which in turn means an increased SO<sub>2</sub> level after the oxidization step. In this way the SO<sub>2</sub> level in the entire system can be significantly increased. This gives the following benefits the SO<sub>2</sub> concentration after the SO<sub>2</sub> scrubber can be:

10

1. Production of a sodiumbisulfite solution with a relative low pH is made possible.
2. The size of the plant can be reduced
3. NO<sub>x</sub> emission is reduced (see above)

15

The first benefit is accomplished since an increased SO<sub>2</sub> level in a gas, from an equilibrium point of view, gives a lower pH in the absorption medium. Since the addition of alkali is reduced a bisulfite solution is formed. This acid can be utilized as acidification in e.g. the bleach plant or the tall oil plant. An increased SO<sub>2</sub> -level in the recirculated gas results however in an increased SO<sub>2</sub> discharge from the system via the bleed off to the atmosphere (10). Connecting a scrubber in this point, to absorb SO<sub>2</sub> can cure this. A scrubber in this position is preferably designed with multiple absorption steps, e.g. of the same design as the stripper. It could be so that only SO<sub>2</sub> is permitted to be absorbed in this position. In that way the SO<sub>2</sub> scrubber (3) can be eliminated from the system.

20

25

The second benefit follows the fact that the circulating gas volume substantially determines the size of the equipment. Since an increased SO<sub>2</sub> content facilitates a higher ratio of condensate/stripper gas flow, the gas flow in the system can be reduced.

30

The cleaned condensate will contain very low levels of sulphides and also any terpenes will be stripped off. This will give a condensate which is rather free from nasty-smelling contaminants. Methanol is another significant contaminant in black liquor condensate.

Some of the methanol will be stripped off in the stripper and some will stay in the condensate. The amount stripped off methanol is dependent on the ratio of supplied condensate to gas and the volume of the circulated gas.

5 The heat economy in the system is excellent since no external heat energy must be added. In the oxidation stage, heat is furthermore generated. This energy can compensate for various energy losses in the system, and any surplus can be absorbed as heat in the outgoing condensate. In other systems, where for example air is used as stripper gas, a significant amount of heat is absorbed in the air since the warm condensate transfers water  
10 vapor in contact with air. This cools down the condensate, which is avoided in the present invention, where any possible evaporated water vapor is returned to the system. It might also be possible to recover heat from the system by implementing a heat exchanger in the system. With such a heat exchanger, which cools the system, the temperature can be controlled.

15 There might also be a need to supply heat to the system. One reason could be to avoid oversaturated gas in certain parts of the system. As the recirculated gas, for instance after the stripper, is saturated with water vapor there is a risk that water droplets will fall out as moisture in the gas. By heating the gas, it would be possible to eliminate that moisture.

20 The investment costs and the size of equipment is mainly directly proportional to the amount of recirculated gas. For that reason it is important to minimize the gas recirculation. This will consequently have an impact on the methanol removal. It is therefore reasonable to count with a certain amount of methanol still remaining in the  
25 condensate. Methanol, as a pollutant in the condensate can be a drawback if the condensate is discharged to the recipient. If the condensate is being recirculated back into the process, e.g. as process water in the bleach plant, brown stock washing or limewashing, then the condensate is excellent in spite of the methanol content.

Methanol has a positive impact on bleaching, it acts as a radical scavenger and it also  
30 increases the solubility of lignin. Furthermore, this condensate is metal free. Normal process water prepared from nearby water streams always contains a certain amount of

metals, such as i.a. transition metals. These transition metals can be very harmful for the bleaching process since they decompose the bleaching agents such as hydrogen peroxide. Since the methanol act as a radical scavenger, the degradation of cellulose molecules will decrease. A metal free condensate used in the bleach plant therefore has significant benefits in spite of a certain methanol content. By recirculating the condensate to the process a discharge of oxygen consuming matters is avoided. The methanol enrichment in the process is very marginal, since the discharge of methanol from the process is relatively large for each process cycle.

10 The stripping of condensate can be performed in several different ways. The type of equipment chosen shall be an equipment having a very high stripper efficiency. Such type of equipment ought to have several equilibrium steps, where the condensate meets a counterflow of gas. Examples on such equipment are columns with trays or packing material. This is well defined in the technical literature, such as i.e. "Perry's Chemical  
15 Engineers' Handbook", MacGraw-Hill Book Company, 1984.

The oxidization process can be done in different ways, but the relatively low concentrations of combustible components require certain prerequisites for this type of process. A relatively high temperature is needed in order to oxidize the combustible  
20 components. A regenerative thermal oxidization process (RTO) is preferred, where the gas is treated in a heat exchanger under such temperature conditions that almost a complete oxidization takes place. Example on such a process is described in the patent application PCT/SE85/00257.

25 Scrubbing of the  $\text{SO}_2$  gas can be done with an alkaline solution. At a pulp mill there is a surplus of alkaline process fluids. One such fluid is oxidized white liquor. In the oxidized white liquor the sulfides have been removed by oxidization. White liquor is such a strong alkali that  $\text{SO}_2$  easily can be absorbed. One equilibrium stage is sufficient. A venturi scrubber is a piece of equipment wherein one equilibrium stage is almost achieved. A  
30 relatively high gas velocity can be maintained in a venturi scrubber, which makes it compact. The scrubber medium is circulated through the venturi.

The pH of the scrubber medium shall be controlled in order to control the SO<sub>2</sub> level in the gases leaving the scrubber. The venturi scrubber has also a significant benefit in that the circulating liquid can have a relatively short residence time. This implies a fast control of the pH in the scrubber. As the scrubber has only almost one equilibrium stage instead of several, a rapid response time is also achieved.



## CLAIMS

1. A method of removing sulphides and other volatile contaminants from liquor vapor condensate from a pulp manufacturing process,

5 c h a r a c t e r i z e d t h e r e i n,

that the said liquor vapor condensate is fed into a stripper (1), which is part of a closed loop comprising said stripper (1) and a regenerative thermal oxidization process (RTO)(2), in which loop a gas (4), primarily consisting of air and such components formed or stripped off in the loop, is circulated, and where the circulating gas is stripping off sulphides and  
10 other volatile components from the liquor vapor condensate (5), whereafter the gas stream (6) is fed into a RTO-process (2), where the stripped off components are combusted under formation of  $\text{SO}_2$ , and thereafter is the  $\text{SO}_2$  enriched gas (7) fed either to a  $\text{SO}_2$  scrubber (3), where preferably alkali is used as absorption medium (8), whereafter the circulating gas is returned to the stripper (1).

15

2. A method as claimed in claim 1,

c h a r a c t e r i z e d i n,

that the  $\text{SO}_2$  scrubber (3) is part of the closed loop.

20 3. A method as claimed in claims 1 or 2,

c h a r a c t e r i z e d i n,

that a minor portion of the gas (10) is bled off from the loop, at the same time air or some other oxygen containing gas (9) is supplied, to ensure that sufficient oxygen is present to safeguard that the oxidization in the RTO-process (2) takes place.

25

4. A method as claimed in anyone of the preceding claims,

c h a r a c t e r i z e d i n,

that the alkali (8) used as absorption medium is oxidized white liquor.

30

5. A method as claimed in anyone of the preceding claims,  
characterized in,  
that the degree of acidification in the SO<sub>2</sub> scrubber (3) is controlled to ensure sufficient  
amount of SO<sub>2</sub> remaining in the gas (4) when it is returned to the stripper (1§), where SO<sub>2</sub>  
5 acidifies the condensate (5) and thereby contributes to enhance the stripping off of  
sulphides from the condensate.

6. A method as claimed in claim 1,  
characterized in,  
10 that a heat exchanger is installed at a suitable place in the closed loop, to recover or supply  
energy and thereby to control the temperature in the system.

7. A method as claimed in claim 1,  
characterized in,  
15 that the amount of recirculated gas versus the amount of condensate is controlled for the  
purpose of optimizing the methanol content in the condensate.

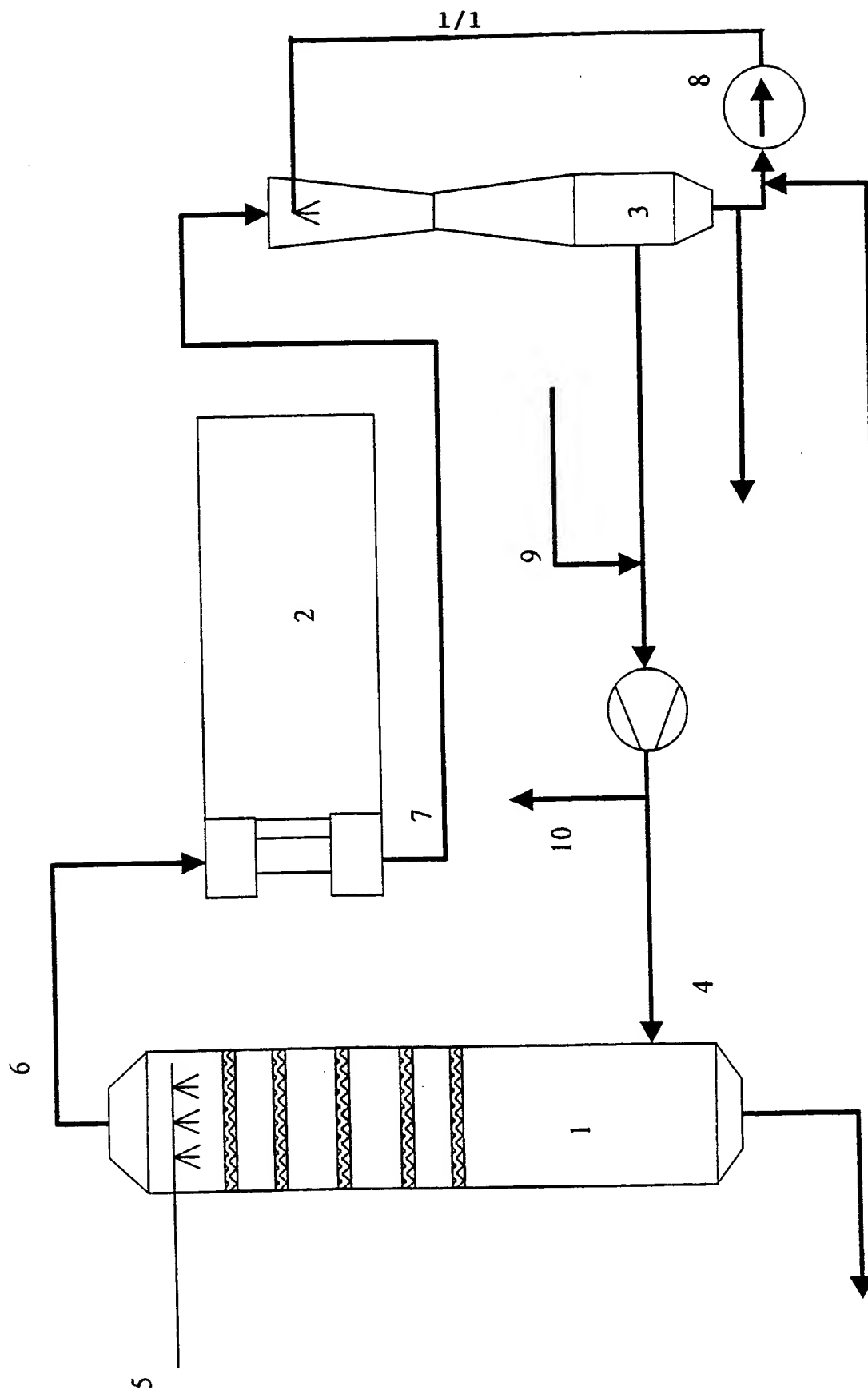
8. A method as claimed in claim 7,  
characterized in,  
20 that such condensate is used as process water in the bleach plant to reduce the bleaching  
chemical cost.

9. A method as claimed in claim 1,  
characterized in,  
25 that the gas (10) being bled off from the system is minimized by using pure oxygen or  
an oxygen enriched air mixture, necessary as make up gas (9) for the oxidization.

10. A method as claimed in claims 1 or 2,  
characterized in,  
30 that the bled off gas (10) from the system is scrubbed with regard to SO<sub>2</sub> in a separate  
scrubber, which preferably is made up of several absorption steps.

11. A method as claimed in anyone of the preceding claims,  
characterized in,  
that the SO<sub>2</sub> level is raised to such a level in the system that the absorption medium in the  
SO<sub>2</sub> scrubber gets sufficient acidic, so that this fluid can be utilized as acidification agent  
5 in other areas of the pulp mill, e.g. the bleach plant or the tall oil plant.

FIG 1



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## A. CLASSIFICATION OF SUBJECT MATTER

IPC7: D21C 11/08, D21C 11/06

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

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SE,DK,FI,NO classes as above

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EDOC, WPI, FULLTEXT

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 1354499 A (CHEMECH ENGINEERING LTD.), 30 May 1974 (30.05.74) --	1-11
A	WO 8600389 A1 (HEED, BJÖRN), 16 January 1986 (16.01.86) --	1-11
P,A	WO 9855685 A1 (THERMATRIX INC.), 10 December 1998 (10.12.98) -- -----	1-11

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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**INTERNATIONAL SEARCH REPORT**  
Information on patent family members

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**PCT/SE 99/02170**

Patent document cited in search report			Publication date	Patent family member(s)		Publication date
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				DE	3590307 T	04/06/87
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				SE	8403330 D	00/00/00
				US	4741690 A	03/05/88
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				EP	0958726 A	24/11/99
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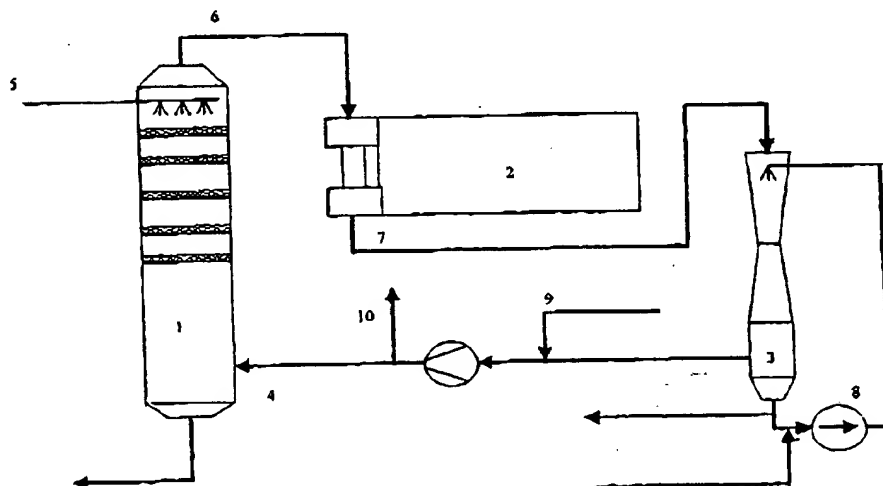
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(54) Title: A METHOD OF CLEANING SULFIDE CONTAMINATED CONDENSATES



## (57) Abstract

A method to remove sulphides and other volatile contaminants from liquor vapor condensate in a pulp manufacturing process, where the mentioned liquor vapor condensate is fed into a stripper, which is part of a closed loop system comprising said stripper, a regenerative thermal oxidization process (RTO) and a SO<sub>2</sub> scrubber, in which loop a gas is circulated, preferably air and such components formed or stripped off, in this loop whereafter the circulating gas is stripping off sulphides and other volatile components from the liquor vapor condensate, whereafter the gas stream is fed into an RTO process, where the stripped off contaminants are combusted and under formation of SO<sub>2</sub> and thereafter the SO<sub>2</sub> enriched gas is led to a SO<sub>2</sub> scrubber, where preferably alkali is used as absorption medium, and thereafter the circulating gas is returned back into the stripper.

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## A method of cleaning sulfide contaminated condensates

In producing chemical pulp according to the Kraft chemical pulp process, waste liquor is produced that is being evaporated prior to burning. During the evaporation process, liquor vapor is stripped off, which in addition to water vapor, also contains certain volatile contaminants. Such contaminants are hydrogen sulfide, methylmercaptan, dimethylsulfide, methanol, terpenes etc. At the evaporation which takes place as a so called multiple effect evaporation with a number of stages, effects (normally 4 - 7), the liquor vapor is also condensed in multiple stages, whereby also large amounts of the volatile contaminants will condense. The condensation takes place in at least as many stages there are effects. This means that the quality of the condensate varies significantly from the different stages of the evaporation. Normally 2-3 different condensate qualities are being separated, where each one is a mixture of condensates from a number of effects. The dirtiest condensate, (foul condensate), is normally treated in a steam stripper where the volatile components are flashed off. This foul condensate is typically a small amount of the total condensate flow and therefore the steam economy is not affected to any higher degree of the fact that steam is used as the stripper gas. The investment cost can also be kept at a minimum.

The purity of the other condensate qualities is highly dependent on the amount of foul condensate. If the amount of foul condensate is increased the contaminated condensates will be cleaner. A too high amount of foul condensate however the operating and investment cost for the steam stripper system will increase.

The other, less contaminated condensates can to a limited extent be used as process water in dependency of their cleanliness. However if the condensate is too contaminated it can not be re-used but must instead be discharged to the recipient subsequent to some form of treatment.

The primary limiting factor for the use of the contaminated condensate as process water is the content of sulfides, as these can give an unpleasant smell and taste to the pulp. It also creates a significant problem for the working environment. Also terpenes give a smell.

The terpenes however are normally present at very low amounts in the less contaminated condensates.

The technology available to clean these condensates is predominately steam stripping.

5 Since the various condensate flows are very large, the size of the stripper will be significant and a large amount of steam will be required for stripping. The steam volumes will be so large that it will definitely not be economical to use fresh steam. On the other hand it is possible to use flash steam driven off from the evaporation of the waste liquor, in multiple effect evaporation for the stripping. The steam leaving the stripper then can be regained as  
10 heat in the next evaporation effect. The cleaning efficiency of such a stripper is however limited since the flash steam from the preceding effect is already contaminated with sulfides, which limits the degree of purity of the output condensate. Primarily the cleanliness is limited regarding sulphides, as the waste liquor can have a considerable content of sulphides. This sulphide content is dependent on that steam is normally taken  
15 from the first effect, where the temperature is rather high, which gives an increased sulphide content.

Another drawback is that when the steam passes through the stripper, it loses pressure and volatile components are enriched. These two things will reduce the condensation  
20 temperature, which means that the temperature difference available at the evaporation is reduced. The energy and capital cost are both negatively impacted thereby. Furthermore the evaporation plant and the stripper are completely integrated, whereby these two parts can not be independently operated.

25 The dimensions of the stripper also will become large, which means significant costs for the equipment.

In a conventional steam stripper also other volatile components, such as methanol, are stripped off.

30 Air can be used to in lieu of steam to strip the condensates. A big drawback with this method is that air is being contaminated and must be cleaned in some way. The air

volumes can also be very large. Additionally the condensate is being cooled down by the air, which has a lower wet bulb temperature as compared to the temperature of the condensate. For these reasons pure air stripping is not a realistic alternative for a modern and environmentally friendly pulp mill.

5

10

The present invention provides a possibility to strip off primarily sulfides at a very high efficiency from liquor-steam condensates from a pulp manufacturing process, and simultaneously to take care of the sulphur, thus that it will not contaminate the environment. This is being done in a closed loop concept that is comprised of three process steps, where the sulfides are stripped off from the condensate, the stripped off sulfides are being oxidized to sulphur dioxide, and to absorb the sulphur dioxide formed.

The three process steps are consequently:

15

1. Stripping off sulphides from liquor-steam condensate
2. Oxidation of combustible components such as sulphides and hydro carbons.
3. Absorption of sulphur dioxide.

20

By integrating these three process steps (1, 2, and 3) in a closed loop cycle, the cleaning of condensates can be done with a high efficiency, good heat economy, and minimal impact on the environment

25

The invention will in the following text be exemplified with reference to a scheme shown in the attached drawing, which schematically shows the various process steps in accordance with the invention.

30

In the present invention a gas is used as a medium for stripping off the sulphides from the condensate. This gas is substantially and preferably composed of air. This process step is normally designed as a scrubber column 1, where the gas 4 is introduced in the lower section and the condensate 5 in the upper section, thus that the gas and the condensate meet in counterflow contact. The contact means in the scrubber can be trays or packing material. The gas 6 leaving the scrubber will contain sulphides in form i.a. of hydrogen

5 sulphide and methyl mercaptan, but also organic compounds such as methanol and terpenes. This contaminated gas 6 is led to an oxidization process 2, where the gas is treated counterflow in a regenerative heat exchanger. The gas 7 from the oxidization step contains partly sulphur dioxide. These gases are then fed to a contact device, in form of a SO<sub>2</sub> scrubber 3, where the sulphur dioxide is absorbed in a preferably alkaline solution 8. The gas is then returned to the condensate scrubber to be used again as a stripping medium. In this manner is formed a closed the loop. Since oxidation in the closed loop consumes oxygen is necessary to add fresh oxygen. Additional oxygen can be added by supply 9 preferably of air or some other oxygen containing gas. The system does not allow for gas accumulation in the loop and therefore a minor portion of the gas 10 must be bled off. The gas circulation through the three process steps is accomplished by the use preferably of a fan.

15 Since the gas in the closed loop is primarily being circulated, an elevated level of various gas components can accumulate to rather high levels. However, since only a minor portion of the gas is bled off, the discharge of components harmful to the environment, will be limited, in spite of high concentrations in the system.

20 A method of improving the cleaning of the condensate in the stripper is to increase the level of SO<sub>2</sub> after the SO<sub>2</sub> scrubber (3). Such a method will result in that the condensate in the stripper (1) will get a lower pH value. A lower pH value in turn gives a better stripping of sulphides and makes possible an almost complete stripping of sulphides. This would otherwise be difficult to achieve since the condensate contains a smaller amount of alkali components, i.e. ammonia, which would increase the pH value of the condensate when the acidic sulfides are stripped off. An alkali component such as ammonia will remain in the condensate at a lowered pH. Thereby is avoided discharge of ammonia, which should otherwise be transformed to Nox, after the oxidation process.

30 An increase of the SO<sub>2</sub> concentration after the SO<sub>2</sub> scrubber (3) can be obtained by adjusting the supply of alkali to this stage thus that the te absorption medium will get a

comparatively lower pH. The lower the pH the higher the  $\text{SO}_2$  concentration in the gas leaving the scrubber (3). The higher the  $\text{SO}_2$ -level in the gas, which constitutes the stripper media, the better the efficiency of stripping off sulphides from the condensate. In turn this effect can be utilized in such a way that the ratio between the condensate flow and stripper gas flow can be increased with continuous good sulphide stripping. This in turn implies an elevated level of sulphides in the stripper off gases, which in turn means an increased  $\text{SO}_2$  level after the oxidization step. In this way the  $\text{SO}_2$  level in the entire system can be significantly increased. This gives the following benefits the  $\text{SO}_2$  concentration after the  $\text{SO}_2$  scrubber can be:

1. Production of a sodiumbisulfite solution with a relative low pH is made possible.
2. The size of the plant can be reduced
3.  $\text{NO}_x$  emission is reduced (see above)

The first benefit is accomplished since an increased  $\text{SO}_2$  level in a gas, from an equilibrium point of view, gives a lower pH in the absorption medium. Since the addition of alkali is reduced a bisulfite solution is formed. This acid can be utilized as acidification in e.g. the bleach plant or the tall oil plant. An increased  $\text{SO}_2$ -level in the recirculated gas results however in an increased  $\text{SO}_2$  discharge from the system via the bleed off to the atmosphere (10). Connecting a scrubber in this point, to absorb  $\text{SO}_2$  can cure this. A scrubber in this position is preferably designed with multiple absorption steps, e.g. of the same design as the stripper. It could be so that only  $\text{SO}_2$  is permitted to be absorbed in this position. In that way the  $\text{SO}_2$  scrubber (3) can be eliminated from the system.

The second benefit follows the fact that the circulating gas volume substantially determines the size of the equipment. Since an increased  $\text{SO}_2$  content facilitates a higher ratio of condensate/stripper gas flow, the gas flow in the system can be reduced.

The cleaned condensate will contain very low levels of sulphides and also any terpenes will be stripped off. This will give a condensate which is rather free from nasty-smelling contaminants. Methanol is another significant contaminant in black liquor condensate.

Some of the methanol will be stripped off in the stripper and some will stay in the condensate. The amount stripped off methanol is dependent on the ratio of supplied condensate to gas and the volume of the circulated gas.

5 The heat economy in the system is excellent since no external heat energy must be added. In the oxidation stage, heat is furthermore generated. This energy can compensate for various energy losses in the system, and any surplus can be absorbed as heat in the outgoing condensate. In other systems, where for example air is used as stripper gas, a significant amount of heat is absorbed in the air since the warm condensate transfers water vapor in contact with air. This cools down the condensate, which is avoided in the present invention, where any possible evaporated water vapor is returned to the system. It might also be possible to recover heat from the system by implementing a heat exchanger in the system. With such a heat exchanger, which cools the system, the temperature can be controlled.

15 There might also be a need to supply heat to the system. One reason could be to avoid oversaturated gas in certain parts of the system. As the recirculated gas, for instance after the stripper, is saturated with water vapor there is a risk that water droplets will fall out as moisture in the gas. By heating the gas, it would be possible to eliminate that moisture.

20 The investment costs and the size of equipment is mainly directly proportional to the amount of recirculated gas. For that reason it is important to minimize the gas recirculation. This will consequently have an impact on the methanol removal. It is therefore reasonable to count with a certain amount of methanol still remaining in the condensate. Methanol, as a pollutant in the condensate can be a drawback if the condensate is discharged to the recipient. If the condensate is being recirculated back into the process, e.g. as process water in the bleach plant, brown stock washing or limewashing, then the condensate is excellent in spite of the methanol content.

25 Methanol has a positive impact on bleaching, it acts as a radical scavenger and it also increases the solubility of lignin. Furthermore, this condensate is metal free. Normal process water prepared from nearby water streams always contains a certain amount of

30

metals, such as i.a. transition metals. These transition metals can be very harmful for the bleaching process since they decompose the bleaching agents such as hydrogen peroxide. Since the methanol act as a radical scavenger, the degradation of cellulose molecules will decrease. A metal free condensate used in the bleach plant therefore has significant benefits in spite of a certain methanol content. By recirculating the condensate to the process a discharge of oxygen consuming matters is avoided. The methanol enrichment in the process is very marginal, since the discharge of methanol from the process is relatively large for each process cycle.

The stripping of condensate can be performed in several different ways. The type of equipment chosen shall be an equipment having a very high stripper efficiency. Such type of equipment ought to have several equilibrium steps, where the condensate meets a counterflow of gas. Examples on such equipment are columns with trays or packing material. This is well defined in the technical literature, such as i.e. "Perry's Chemical Engineers' Handbook", MacGraw-Hill Book Company, 1984.

The oxidization process can be done in different ways, but the relatively low concentrations of combustible components require certain prerequisites for this type of process. A relatively high temperature is needed in order to oxidize the combustible components. A regenerative thermal oxidization process (RTO) is preferred, where the gas is treated in a heat exchanger under such temperature conditions that almost a complete oxidization takes place. Example on such a process is described in the patent application PCT/SE85/00257.

Scrubbing of the  $\text{SO}_2$  gas can be done with an alkaline solution. At a pulp mill there is a surplus of alkaline process fluids. One such fluid is oxidized white liquor. In the oxidized white liquor the sulfides have been removed by oxidization. White liquor is such a strong alkali that  $\text{SO}_2$  easily can be absorbed. One equilibrium stage is sufficient. A venturi scrubber is a piece of equipment wherein one equilibrium stage is almost achieved. A relatively high gas velocity can be maintained in a venturi scrubber, which makes it compact. The scrubber medium is circulated through the venturi.

The pH of the scrubber medium shall be controlled in order to control the  $\text{SO}_2$  level in the gases leaving the scrubber. The venturi scrubber has also a significant benefit in that the circulating liquid can have a relatively short residence time. This implies a fast control of the pH in the scrubber. As the scrubber has only almost one equilibrium stage instead of several, a rapid response time is also achieved.



## CLAIMS

1. A method of removing sulphides and other volatile contaminants from liquor vapor condensate from a pulp manufacturing process,

5 characterized therein,

that the said liquor vapor condensate is fed into a stripper (1), which is part of a closed loop comprising said stripper (1) and a regenerative thermal oxidization process (RTO)(2), in which loop a gas (4), primarily consisting of air and such components formed or stripped off in the loop, is circulated, and where the circulating gas is stripping off sulphides and other volatile components from the liquor vapor condensate (5), whereafter the gas stream  
10 (6) is fed into a RTO-process (2), where the stripped off components are combusted under formation of  $\text{SO}_2$ , and thereafter is the  $\text{SO}_2$  enriched gas (7) fed either to a  $\text{SO}_2$  scrubber (3), where preferably alkali is used as absorption medium (8), whereafter the circulating gas is returned to the stripper (1).

15 2. A method as claimed in claim 1,

characterized in,

that the  $\text{SO}_2$  scrubber (3) is part of the closed loop.

20 3. A method as claimed in claims 1 or 2,

characterized in,

that a minor portion of the gas (10) is bled off from the loop, at the same time air or some other oxygen containing gas (9) is supplied, to ensure that sufficient oxygen is present to safeguard that the oxidization in the RTO-process (2) takes place.

25 4. A method as claimed in anyone of the preceding claims,

characterized in,

that the alkali (8) used as absorption medium is oxidized white liquor.

5. A method as claimed in anyone of the preceding claims,  
characterized in,

that the degree of acidification in the SO<sub>2</sub> scrubber (3) is controlled to ensure sufficient amount of SO<sub>2</sub> remaining in the gas (4) when it is returned to the stripper (1§), where SO<sub>2</sub> acidifies the condensate (5) and thereby contributes to enhance the stripping off of sulphides from the condensate.

6. A method as claimed in claim 1,  
characterized in,

that a heat exchanger is installed at a suitable place in the closed loop, to recover or supply energy and thereby to control the temperature in the system.

7. A method as claimed in claim 1,  
characterized in,

that the amount of recirculated gas versus the amount of condensate is controlled for the purpose of optimizing the methanol content in the condensate.

8. A method as claimed in claim 7,  
characterized in,

that such condensate is used as process water in the bleach plant to reduce the bleaching chemical cost.

9. A method as claimed in claim 1,  
characterized in,

that the gas (10) being bled off from the system is minimized by using pure oxygen or an oxygen enriched air mixture, necessary as make up gas (9) for the oxidization.

10. A method as claimed in claims 1 or 2,  
characterized in,

that the bled off gas (10) from the system is scrubbed with regard to SO<sub>2</sub> in a separate scrubber, which preferably is made up of several absorption steps.

11. A method as claimed in anyone of the preceding claims,

characterized in,

that the  $\text{SO}_2$  level is raised to such a level in the system that the absorption medium in the  $\text{SO}_2$  scrubber gets sufficient acidic, so that this fluid can be utilized as acidification agent

5 in other areas of the pulp mill, e.g. the bleach plant or the tall oil plant.

FIG 1

